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# THE ANALYST

## PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held on Wednesday, March 7th, at the Chemical Society's Rooms, Burlington House. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

Certificates were read for the first time in favour of Messrs. John Myers, F.I.C., and John Loudon Buchanan, F.I.C.

Certificates were read for the second time in favour of Messrs. Joseph John Valentine Backes, A.R.C.Sc., A.I.C., D.I.C., Samuel Gordon Stevenson, A.I.C., Laurence Barnett Timmis, M.Sc.Tech. (Manc.), A.I.C., Richard William Sutton B.Sc. Tech. (Manc.), A.I.C., Alfred Edward Johnson, B.Sc. (Lond.), F.I.C., A.R.C.S.I., Ernest Victor Jones, F.I.C., Francis Kenelm Donovan, B.Sc., and S. Gordon Liversedge, F.I.C.

The following were elected members of the Society:—Messrs. George Henry Appleyard, F.I.C., James Walter Black, B.Sc. (Lond.), Arthur William Starey, A.R.C.S., B.Sc. (Lond.), A.I.C., and John Matthew Wilkie, B.Sc. (Lond.), F.I.C.

The following papers were read:—"The Examination of Firearms and Projectiles in Forensic Cases," by A. Lucas, O.B.E., F.I.C.; "The Interpretation of the Results obtained in the Analysis of Potable Waters," by R. C. Frederick; and "Determination of the Purity of Vanillin," by Sydney B. Phillips, A.I.C.

## Obituary.

### FREDERICK JAMES LLOYD.

Born in 1852, and associated as a young man with the late Dr. Augustus Voelcker Frederick James Lloyd was for a long period a well-known figure in chemical, and especially in agricultural, circles. Though never occupying an appointment as Public Analyst under the Sale of Food and Drugs Act, he held posts as Agricultural Analyst under the Fertilisers and Feeding Stuffs Act, becoming early a member of our Society and serving on its Council, 1903–4.

Coming up to London in 1870 from Bristol Grammar School, where he had H. H. B. Shepherd as his companion, he became assistant in Dr. Voelcker's private laboratory, having as fellow-workers there Dr. Bernard Dyer and Mr. Alfred Smetham, both Past-Presidents of our Society. After that he was successively assistant to Dr. Thos. Stevenson at Guy's Hospital, and senior assistant in the laboratory of the Royal Agricultural Society of England, of which Society his cousin, the late Mr. H. M. Jenkins, was then secretary. In 1884 he started practice on his own account in Lombard Court, City, and this practice, though it never reached any material dimensions, he carried on, with several changes, till his death.

It was chiefly in connection with dairy matters that Lloyd was known, and in this connection he did really good work alike for the Bath and West of England Agricultural Society and for the British Dairy Farmers' Association, of which latter body he became consulting chemist in 1885. In this capacity he contributed a number of articles and reports to the Journal of the Association, and will perhaps be best known for the laborious work that devolved on him in connection with the milking tests at the annual shows of the Association held at the Royal Agricultural Hall. From small beginnings, these, by their increasing popularity, grew into a very big undertaking, of which, as regards the analyses and awards, Lloyd had the sole charge.

His best scientific work was, however, done in connection with investigations carried out for the Bath and West of England Agricultural Society upon Cheddar cheese-making, and on the scientific side of which his researches threw much light. Similarly, he was for a considerable time engaged in research on cider-making, and it is not too much to say that he was the first to put this industry on something approaching a scientific basis. For some years he acted as Abstractor for the Journal of the Chemical Society, and was a familiar figure and frequent speaker at the Central Chamber of Agriculture, the Farmers' Club, etc.

Lloyd was a careful analyst, a well-read man, and hard worker, but, though he did good work, it cannot be said that it was sufficiently appreciated or that he reaped the benefits from it that he should have gained. The Dairying industry, on which he mainly relied, never showed itself generous in the matter of professional fees, or in appreciation of chemical assistance, and the closing years of Lloyd's career were attended by many difficulties and anxieties, including at least two forced removals of his laboratory to fresh quarters.

For many of his difficulties he was in great measure himself responsible, peculiarities of manner, together with a somewhat dogmatic way of expressing himself, not helping to ingratiate him with the public whom he desired to serve. In scientific groups his actions and utterances were marked by the same characteristics, and, though he was actuated by the best of motives, and often was able to contribute usefully from his experience to the work of organisations and committees on which he served, he was generally regarded as rather an "awkward" member, and as one who was too much given to the persistent urging of his own particular "hobbies."

But, while saying this, it must be remembered of him that no man had a

higher sense of what constituted honourable professional conduct, and he was ever ready to maintain the dignity of the chemical profession, and the claims it had for recognition by the official world and the public generally. Moreover, he was always insistent on the great benefits that would accrue to our industries, and chiefly to agricultural industries, by making a wider use of the lessons and guidance of science. In him one must recognise an honest worker, and one who, in spite of certain drawbacks, was a man to be esteemed, and in whom many of us have lost a good friend.

He died in London on February 8th, after an illness of only three days, at the age of 70. The funeral, on February 13th, was attended by Dr. Dyer and Mr. E. W. Voelcker.

J. A. VOELCKER.

# Some Observations with Regard to the Unsaponifiable Matter and Sterols of Edible Fats.

BY DAN. W. STEUART, B.Sc.

(*Read at the Meeting, December* 6, 1922.)

I. STEROLS. The following analytical data exemplify a type of margarine which is not easy to analyse with any degree of certainty:—X. MARGARINE (Samples 28 and 29).—Iodine value, 64·3; melting point, 32° C.; saponification value, 195; ether-insoluble bromides, 0; unsaponifiable matter, 0·56 per cent.; Blichfeldt-Gilmour distillation, T = 1·0; butter, coconut and palm kernel fats, absent; sesame oil, absent; cottonseed oil, present; nickel, absent, *i.e.* not one part per million.

These tests only prove definitely the presence of cottonseed oil; animal fats are probably present, but palm oil and hardened oils are by no means excluded.

A few experiments have been made to see whether in such a case as this, an estimation and examination of the sterols would yield any additional information.

An examination of the sterol prepared from a sample of fat will show definitely whether the fat is of purely animal origin or whether vegetable fat is present. The results of this work indicate that the sterol acetate examination cannot be used to demonstrate the presence of animal fats in mixtures containing vegetable oils. Marcusson and Meyerheim (1916) found from 8 to 14 per cent. of sterol in the unsaponifiable matter of animal fats, *e.g.* cod-liver oil and tallow, and 33 to 55 per cent. in that of vegetable oils. This suggested that the proportion of sterol in the unsaponifiable matter of a margarine fat might give some indication as to its origin; but such a hope can no longer be entertained.

The results of the tests are shown in the accompanying table. Samples 28 and 29 refer to X. margarine, the sterol content of which amounted to 0·15 or 0·16 per cent., and formed about 26 per cent. of the unsaponifiable matter. In

the animal fats the sterol content varied from 0·04 per cent. in beef stearin to 0·22 per cent. in New Zealand butter. In the vegetable fats it varied from 0·03 per cent. in palm oil to 0·58 per cent. in maize oil. It constituted only 7 per cent. of the unsaponifiable matter of palm oil, but 48 per cent. of it in maize oil.

Hydrogenation (hardening) decreases the sterol content of an oil. Marcusson and Meyerheim on progressively hardening a marine animal oil obtained 0·13, 0·10, 0·07, 0·05, and 0·02 per cent. of sterol. A similar decrease was found here in the case of a vegetable oil. Arachis oil gave 0·20 per cent. of sterol, two partially hardened samples gave 0·16 and 0·14 per cent. respectively, and a fully hardened sample gave 0·10 of sterol.

ESTIMATION AND EXAMINATION OF STEROL.—The unsaponifiable matter from 50 grms. of fat was warmed with 50 c.c. of 95 per cent. alcohol and mixed with 50 c.c. of hot 90 per cent. alcohol containing 0·5 to 1 grm. of digitonin. After standing overnight the precipitate of digitonin steride was filtered off, washed on the filter paper with 95 per cent. alcohol and with ether and weighed after drying at 110° C. Per cent. of sterol = (Wt. + 0·014) × 0·243 × 2. The steride was then transferred to a small beaker and about 1 c.c. of acetic anhydride added per 0·1 grm. of steride, the beaker covered with a watch glass, and the liquid boiled until the steride had dissolved. On cooling, nearly all the sterol acetate crystallised out (Olig, *Zeitsch. Nahr. Genussm.*, 1914, **28**, 129). This was filtered off, and the crystals dried with the aid of suction, dissolved through the filter paper with ether, and, after the ether had evaporated, dissolved in absolute alcohol. As the crystals of acetate separated out they were filtered off, and another or several crops thus taken from the alcohol. The wet crystals, thrown on a porous plate, were kept overnight at 37° C. before the melting points were determined. These are shown in column A. In some cases on boiling the acetic anhydride mother liquor down to half the previous volume and cooling, another crop of crystals was obtained. This was worked up as before and the melting point taken (see column B). In some cases fractions were recrystallised from alcohol as shown in the table.

In the case of sample 30, the unsaponifiable matter was acetylated directly, and the acetate which crystallised out was worked up in the usual way. This sample consisted of vegetable oil containing polymerised oil, the refractive index being high in relation to the iodine value. (*Cf.* Lund, *Zeitsch. Nahr. Genussm.*, 1922, **44**, 175).

Duplicate sterol estimations agreed closely, *e.g.* samples 23 and 24, and 28 and 29. Also, when a considerable range of samples had been examined two mixtures of these were made and the sterol estimated in the mixtures with the following results:

| No. 26 | | | No. 27 | | |
|---|---|---|---|---|---|
| | Per Cent. | Grm. | | Per Cent. | Grm. |
| Lard | 25 | = 0·032 | Palm | 40 | = 0·012 |
| Oleo | 40 | = 0·048 | Hardened seal | 20 | = 0·012 |
| Jus | 10 | = 0·009 | Jus | 10 | = 0·009 |
| Cotton oil | 15 | = 0·035 | Cotton oil | 10 | = 0·023 |
| Arachis oil | 10 | = 0·020 | Hardened arachis | 20 | = 0·032 |
| | 100 | = 0·144 calculation | | 100 | = 0·088 |
| | | = 0·14 analysis | | | = 0·09 |

The sterol acetate from the animal fats melted at 114° to 114·5° C. Only in the case of the hardened whale oil did it melt at a lower temperature. On fractionating the acetates the fractions melted at practically the same temperature, *i.e.* the melting point of cholesterol acetate.

In the case of vegetable fats the melting point of the sterol acetate varies, and on fractionating the acetate the fractions melt at different temperatures. The sterol of vegetable fats is not a pure substance but a mixture. The fractions from rape oil melted at 128° to 139° C. Pure sitosterol acetate melts at 127° C. (Jäger and Klamroth), and stigmasterol acetate at 141° C. (Windaus and Hauth). In the case of cottonseed oil the fractions melt between 114° and 125° C. The figure for the lower fractions suggests the presence of cholesterol* in the oil. The low melting point does not seem to be due to digitonin or other impurities. In the case of cottonseed oil (samples 23 and 24) the dried digitonin steride from 100 grms. oil was thoroughly extracted with ether in a Soxhlet apparatus before acetylation, and the solution of acetates in acetic anhydride was thrown into 160 c.c. of water, 40 c.c. of alcohol added, and two drops of phenolphthalein solution, and then 50 per cent. sodium hydroxide solution, drop by drop, to neutrality. The solution was next extracted with ether, and the extract washed repeatedly with 20 per cent. alcohol. On evaporating the ether the sterol acetate separated out, and was then worked up in the usual way. According to Lifschütz (1918), the sterol acetate dissolves in ether, while digitonin remains behind in dilute alcohol. In the case of the sesame oil (sample 13), the digitonin steride was boiled with 50 c.c. of xylene; this brought the sterol into solution and left the digitonin undissolved. The sterol was then acetylated and worked up in the usual way. In the case of cottonseed oil (sample 22), the unsaponifiable matter from 100 grms. was dissolved in petroleum spirit of low b.pt. and cooled in the ice safe until a considerable amount of the unsaponifiable matter separated out; this was then filtered off. The two fractions separated in this way (Lewkowitsch) were treated with digitonin separately, but no sharp separation of the sterols was thus effected.

The figures give little information with regard to hydrogenation on the melting point of the sterol acetates. In the case of hardened seal oil the usual cholesterol acetate melting point was obtained. In the case of arachis oil, hardening caused a slight rise in the melting point of the sterol acetate. In the case of cottonseed oil an old sample of fully hardened oil gave sterol acetates with lower melting points than those obtained from fresh cottonseed oil. Conceivably age (oxidation?) effects some change in the sterol, causing a lowering of the melting points of the acetate; hence, in the case of the older samples of fat, the age has been noted. (*Cf.* Polenske, *Arb. Kaiserl. Gesundh*, 1912, **38**, 402).

The X. margarine samples 28 and 29 yielded sterol acetates melting between 128 and 122° C. The mixtures containing 75 per cent. of animal fat (No. 26)

* This sterol is not cholesterol, however. Cholesterol melts at 148° C., and sitosterol (the chief phytosterol of vegetable fats) at 139° C., whilst the sterol prepared from the low-melting acetates of cottonseed oil melted at 133° C. (*Cf.* Wagner and Clement, *Zeitsch. Nahr. Genussm.*, 1909, **17**, 267).

and 30 per cent. of animal fat (No. 27) yielded similar melting points for similar fractions.

The sterol acetates prepared from some vegetable oils contain fractions of lower melting point, which makes it impossible to detect the presence of animal fats in mixtures containing such vegetable fats by an examination of the sterol acetates.

II. LECITHIN.—It is commonly stated (*e.g.* by Lewkowitsch) that the high unsaponifiable matter of maize oil is due to lecithin. As lecithin is a saponifiable compound, this matter seemed worthy of further examination.

| | Unsap. matter Per Cent. | Sterols Per Cent. | Sterols in unsap. matter Per Cent. | Melting Points °C. A | B |
|---|---|---|---|---|---|
| *Animal Fats.* | | | | *Cholesterol Acetate.* | |
| 1. Beef—jus. | 0·52 | 0·09 | 18 | 114 | |
| 2. —oleo | 0·37 | 0·12 | 32 | 114·5 | 114·5 |
| 3. —stearin | 0·31 | 0·04 | 12 | 114·5 | |
| 4. Lard | 0·34 | 0·13 | 38 | 114<br>114 114 | 114 |
| 5. Seal oil | 0·36 | 0·06 | 16 | 114 | |
| 6. Hardened seal, m.pt. 38° C. | 0·31 | 0·05 | 16 | 114 | |
| 7. Hardened whale, m.pt. 38° C. | 1·07 | 0·10 | 9 | below 114 | |
| 8. Butter fat | — | 0·22 | — | 114 114 | |
| *Vegetable Fats.* | | | | *Phytosterol Acetates.* | |
| 9. Palm oil. C. | 0·37 | 0·03 | 7 | 127 | |
| 10. Palm kernel | — | 0·06 | — | 128–128–125·5 | |
| 11. Coconut | — | 0·06 | — | 127 | |
| 12. Soya bean oil (1918) | 0·84 | 0·23 | 27 | 133–132–129<br>133 | |
| 13. Sesame oil | 1·52 | 0·43 | 28 | 125–122 | |
| 14. Linseed oil (old) | 0·87 | 0·37 | 41 | 131–129–126<br>132 | 116 |
| 15. Rape oil with paraffin | 9·25 | 0·32 | — | 135–129<br>137 134 128<br>139 | |
| 16. Arachis oil. E. | 0·58 | 0·20 | 34 | 125 | |
| 17. Hardened arachis. D. | 0·35 | 0·16 | 45 | 127 | |
| 18. Hardened arachis. E. | 0·41 | 0·14 | 34 | 127 | |
| 19. Hardened arachis. E. m.pt. 64° C. | 0·45 | 0·10 | 22 | 128 | |
| 20. Maize oil (1914) | 1·21 | 0·58 | 48 | 129–126–119<br>130<br>133 | 124 |
| 21. Cottonseed oil. W.44 | 0·54 | 0·23 | 42 | 125 121<br>125·5–122·5–122·5–118·5 | 115 |
| 22. Cottonseed oil (sol. in petroleum spirit)<br>(insol. in petroleum spirit) | | | | 125–120<br>125–124 | |
| 23. Cottonseed oil. J. cloudy | 0·61 | 0·26 | 43 | 125–124–123–116–114·5–114 | |
| 24. Cottonseed oil. J. filtered | 0·64 | 0·26 | 41 | | |
| 25. Hardened cottonseed oil, m.pt. 62° C. (1911) | 0·56 | 0·23 | 41 | 121–119–117 | |
| *Mixtures and Unknown Samples.* | | | | | |
| 26. 75 per cent. animal fat, 25 per cent. vegetable | 0·43 | 0·14 | 33 | 128–127–123 | |
| 27. 30 per cent. animal fat, 70 per cent. vegetable | 0·35 | 0·09 | 26 | 128 | 123 |
| 28. Margarine | 0·56 | 0·16 | 28 | 128–126–124 | 122 |
| 29. Margarine | — | 0·15 | — | | |
| 30. | 0·77 | — | — | 127–119 | |

The material used was the clear oily liquid filtered from a sample of commercial lecithin. On treatment with acetone this oil yielded 25 per cent. of crude lecithin. This was dried with a solution of magnesium acetate (Grossfeld, 1920), incinerated, and dissolved in dilute hydrochloric acid, and the phosphate estimated as magnesium pyrophosphate. The phosphoric anhydride amounted to 2·02 per cent. of the oil, which was equivalent to 22·3 per cent. of lecithin. The total nitrogen in the oil was 0·420 (duplicate estimation = 0·413 per cent.), which was equivalent to 22·7 of lecithin in the oil. The oil yielded 4·23 per cent. of unsaponifiable matter (sterol acetate, m.pt., 132° C.), in which the total nitrogen amounted to 0·030 per cent., calculated on the oil, corresponding to 1·67 per cent. of lecithin in the oil. It appears, then, that not more than 8 per cent. of the lecithin of the oil could have been present in the unsaponifiable matter in this somewhat extreme case.

Lecithin is used in somewhat minute quantities in some margarines. In such a case it has been detected in 5 grms. of filtered fat by evaporating the fat with magnesium acetate and concentrated acetic acid, incinerating the residue, and precipitating the phosphate as ammonium phospho-molybdate, a control test being made at the same time. In this case the preliminary treatment with acetone is useless. This rough test will detect 0·1 per cent. of lecithin in a fat.

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## DISCUSSION.

Dr. J. C. DRUMMOND enquired if the author had any observations to make as regards the constituents—other than sterols—of unsaponifiable matter; such as hydrocarbons and higher alcohols. He also enquired where the author had obtained his digitonin.

Mr. E. HINKS asked whether any effect due to feeding on coconut cake was observable in the test. In a crystallographic test he had worked out some years ago (ANALYST, 1907, 32, 160) a coconut ration did not affect the result in the way the addition of coconut oil to butter-fat did.

Mr. C. A. MITCHELL said he thought that some method might be devised for solving the problem by completely hydrogenating the fat so as to get the fatty acids into their highest possible state of saturation, and then crystallising them from suitable solvents previously saturated with specific pure fatty acids, such as behenic or arachidic acids. The estimation might be made in the same way as in the method devised by Mr. Hehner and him for estimating stearic acid.

Mr. E. R. BOLTON called attention to an experiment in which the constants of butter-fat were affected considerably by feeding a cow upon desiccated coconut containing about 70 per cent. of fat.

Mr. STEUART, replying to Dr. Drummond's question, said that all that was wanted in this particular case was a method of analysing margarine. As to whether the sterols were real entities, he thought it rather doubtful whether pure substances had been obtained in the case of the phytosterols. With regard to coconut oil feeding experiments; more experiments ought to be carried out, and he

considered that to feed a cow for a short time with, say, 1 lb. daily of coconut meal containing 3 per cent. of oil would have a very different effect on the butter from feeding heavily with cake containing 9 per cent. of oil over a long period. He said that the hydrogenation question had already been considered to this extent: that the melting points of practically all the fully hardened edible fats and oils were known, and that the melting point of the fully hardened margarine fat would not throw much light on the original composition of the margarine. The digitonin used had been obtained since the war, it was probably Merck digitonin, and it cost 5s. per grm.

# Note on the Presence of Sulphur Dioxide in Cattle Foodstuffs after Fumigation.

By H. ALAN PEACOCK, B.Sc.

(*Read at the Meeting, December* 6, 1922).

A SAMPLE of cattle cake which had been stored in a building fumigated by burning sulphur (following an outbreak of foot-and-mouth disease) was submitted for analysis with a view to ascertaining if it had absorbed any sulphur dioxide. In this instance no sulphur dioxide was found.

The point was raised, however, as to whether some sulphur dioxide might not originally have been absorbed and, by the time the analysis was made, have disappeared.

Accordingly, as a preliminary experiment, various types of cattle cake, both in their original block form, and in a powdered condition, were placed in a closed cupboard and treated by burning sulphur. The experiment was conducted in much the same way that fumigation would be carried out on a farm, though care was taken to expose the cakes in such a manner that they were all subjected to the same conditions. The material was exposed to the fumes of the burning sulphur for about one hour, the powdered cakes being spread as uniformly as possible on filter papers, 18·5 cm. in diameter, whilst the cakes in blocks were suspended from the top of the cupboard to allow a free circulation of the gas about them.

In the subsequent analyses of the cakes it was considered sufficient to estimate the total amount of reducing substances volatile in steam, the figures obtained from a control experiment being afterwards deducted. Any excess of such substances in the treated, over the untreated, cake, was deemed to be due to the presence of sulphur dioxide.

The method adopted for the estimation of the reducing substances was as follows:—The ground-up cake was slightly acidified with phosphoric acid, and then distilled in steam for twenty minutes, the distillate being collected in a solution of ferric ammonium sulphate. The solution was acidified with excess of dilute sulphuric acid and titrated with standard potassium permanganate, the results being expressed in terms of sulphur dioxide.

In passing, it might be mentioned that authorities differ as to the most satisfactory method of estimating sulphur dioxide. Sweeny, Outcault and Withrow[1] prefer the permanganate to the iodimetric method, whilst Ferguson[2] and Mathieu[3] prefer the iodimetric method. Again, Kühn and Rühle[3] state that sulphur and sulphurous acid are not completely oxidised to sulphuric acid with iodine, and Cazenave[4] says that iodine estimations are not trustworthy, and prefers to oxidise and estimate the sulphur gravimetrically.

Examination of the cakes immediately after fumigation, and after prolonged standing, gave the following results:

| Description of cake | Control. Reducing substances in terms of $SO_2$ | Sulphur dioxide present after exposure | | Days elapsed since exposure of cake to $SO_2$ |
|---|---|---|---|---|
| | | Powders | Blocks | |
| | Per cent. | Per cent. | Per cent. | |
| Coconut .. .. .. .. | 0·019 | 0·110 | | 2 |
| | | — | 0·056 | 6 |
| | | nil | — | 104 |
| | | — | nil | 108 |
| Bombay Cotton .. .. | 0·013 | 0·163 | — | 1 |
| | | — | 0·026 | 6 |
| | | nil | — | 104 |
| | | — | nil | 108 |
| Egyptian Cotton .. .. | 0·013 | 0·116 | — | 1 |
| | | — | 0·010 | 2 |
| | | — | nil | 6 |
| | | 0·026 | — | 104 |
| Semi-decorticated Ground Nut | 0·006 | 0·071 | nil | 2 |
| | | 0·009 | — | 104 |
| | | — | nil | 108 |
| Linseed .. .. .. .. | 0·013 | 0·093 | 0·030 | 2 |
| | | — | nil | 6 |
| | | nil | — | 105 |
| Palm Kernel .. .. .. | 0·016 | 0·056 | — | 2 |
| | | — | 0·040 | 7 |
| | | nil | — | 105 |
| | | — | nil | 108 |
| Soya .. .. .. .. | 0·009 | 0·075 | — | 2 |
| | | — | nil | 4 |
| | | 0·019 | — | 106 |
| | | — | nil | 108 |

As Mr. A. Chaston Chapman has pointed out,[5] many foods contain volatile reducing substances, and the figures for the control experiment corroborate this, the amounts obtained from similar types of cake agreeing fairly closely with each other. In this connection it may be noted that in the detection of sulphured malt Reinke[6] found that on distilling the malt with phosphoric acid in a current of carbon dioxide, absorbing the sulphurous acid in iodine, and adding barium

chloride, there was no precipitate of barium sulphate, although on making a qualitative test with zinc and sulphuric acid a dark brown stain was given on lead acetate paper, this, apparently, being due to organic bodies.

In connection with the diminution in the amount of sulphur dioxide after standing some days, Bartells Jr.[7] found that when a mixture of sulphur dioxide and air was kept in a carboy containing green vegetation, after a time some sulphur dioxide disappeared. In this instance it was concluded that not only had the vegetation absorbed some of the gas, but also that some of the sulphur dioxide had been oxidised in an excess of moist air.

So far as the experiments have gone it may be concluded that:—(1) Sulphur dioxide may be absorbed by cattle cakes and meals during fumigation, but that after about a week it disappears. (2) The amount of sulphur dioxide absorbed seems to depend on (*a*) the variety of cake; the harder cakes absorbing less than the softer varieties; (*b*) the condition of the foodstuff, *i.e.* whether in block or powder form.

It is well known that poor samples of grain, *e.g.* "foxy" oats, are sometimes treated with sulphur dioxide to improve their appearance. Opinions seem to be divided as to whether such grain is harmful to animals; but in any case it would appear doubtful, in view of the results obtained, whether any deleterious effects could be ascribed to the presence of sulphur dioxide.

## References.

(1) O. R. Sweeny, H. E. Outcault and J. R. Withrow, "The Determination of Sulphur Dioxide," *J. Ind. Eng. Chem.*, 1917, **9**, 949–950.
(2) J. B. Ferguson, "The Iodimetric Determination of Sulphur Dioxide and the Sulphites," *J. Amer. Chem. Soc.*, 1917, **39**, 364–373.
(3) B. Kühn and J. Rühle, "Sulphurous Acid in Chopped Meat," *Zeitsch. Untersuch. Nahr. Genussm.*, 1910, **29**, 10–19.
(4) P. Cazenave, "Estimation of Sulphurous Acid in Wines," *Ann. Falsif.*, 1910, **3**, 154–158.
(5) A. Chaston Chapman, "Examination of Foods for the Presence of Sulphites," Analyst, 1922, **47**, 204–205.
(6) O. Reinke, "Detection of Sulphured Malt," *Chem. Zeit.*, 1910, **34**, 1159.
(7) G. C. Bartells, jun., "Disappearance of Sulphur Dioxide from Dilute Mixtures of Sulphur Dioxide and Air," *U.S. Dept. Int., Bureau Mines Bull.* 98, 1915, 308–323.
(8) L. Mathieu, "Estimation of Sulphurous Acid in Wine," *Ann. Falsif.*, 1910, **3**, 410–417.

## Discussion.

Mr. H. P. Stevens enquired whether there was approximately the same amount of moisture in different varieties of cattle cake.

Mr. Chaston Chapman said that he felt that the main importance of the communication was that it indicated clearly the need for caution on the part of the analyst. As he, himself, and others had shown, it was quite possible to obtain somewhat considerable quantities of barium sulphate resulting from the oxidation of volatile organic sulphur compounds, and not due to sulphur dioxide at all.

The moral was that there was need for great caution in drawing conclusions from the results of the method as ordinarily carried out.

Mr. Chapman referred to the practice of fumigating wine casks by burning sulphur matches, a practice which results in small quantities of sulphur dioxide being communicated to the wine. Cases were on record, however, in which the wine, although originally containing an appreciable quantity of sulphur dioxide, when examined somewhat later was found to yield a negative result owing to the combination of the sulphur dioxide with some of the organic constituents of the wine. He thought that whenever the presence of sulphur dioxide was indicated in the distillation test it was advisable to ascertain whether there was any difference between the result when using hydrogen peroxide on the one hand and bromine on the other as the oxidising agent.

Mr. A. E. PARKES said he also knew of many instances in which the sulphur dioxide contents had disappeared. Sulphur dioxide in some forms was frequently used as a preservative for foodstuffs, and, in his opinion, where preservatives were necessary, perhaps, in moderation, it was one of the least harmful. It was used for the preservation of articles such as lime juice, lemon squash, and sausage meat, &c., but the amount was noticed to decrease after a few days. He thought that in the case of cattle cakes its disappearance might be due to the presence of some oxidising enzymes.

Mr. L. K. BOSELEY said that in samples of preserved red cherries, which he had handled, he had found clear traces of sulphur dioxide and of sulphides; the latter in particular were believed to be deleterious. When preserving these cherries abroad the usual procedure was to place them in small brick chambers, treat with sulphur dioxide until they were white, then with aniline reds, and finally with sugar and glucose in solution.

Mr. E. R. BOLTON referred to the marked action of sulphur dioxide in the process of drying potatoes. Extraordinarily small quantities of sulphur dioxide passed over cut potatoes before drying prevented the discoloration, and yielded an almost white product, and, if the process was not overdone, the excess of sulphate eventually found in the potato was negligible. He also referred to the butcher's custom of dipping a whole carcase into a solution of bisulphite of lime.

Mr. CHAPMAN said that he thought that in the case of potatoes, at any rate, the disappearance of the sulphur dioxide might well be facilitated by the presence of enzymes.

Mr. NORMAN EVERS, reverting to the subject of preserved cherries, said that they were sometimes treated with stannous chloride to fix the colour, with the result that quite considerable quantities of tin were at times found in these cherries.

Mr. CLAREMONT said that the treatment of cattle cakes by fumigation was necessary for the prevention of foot and mouth disease.

Mr. PEACOCK, in reply to the first speaker's question, said that he had not estimated the amount of moisture present in cattle cake, but he thought that all the different varieties had very much the same amount, which was, roughly, 10 per cent. As regards Mr. Chaston Chapman's question, he had formed no conclusive opinion why, or in what form, the sulphur dioxide disappeared.

# Sliding Scales for the Convenient Titration of Strong Liquids by Dilution and Use of Aliquot Parts.

By C. H. D. CLARK, B.Sc., D.I.C., A.I.C.

(*Read at the Meeting, December* 6, 1922.)

## Part II.

By the use of a modification of the sliding scale explained in Part I. (Analyst, 1923, 61), preliminary calculation is avoided, and the reckoning done by the scale itself. This improvement, which involves some slight sacrifice in simplicity, has been introduced by the use of two fresh scales F and G.

A reference to the diagram showing Scale II. will make its construction clear. Scales A, B, C, D, and E have the same significance as in the previous arrangement. This slide rule can best be made by ruling lines upon squared paper, or paper ruled with fairly narrow-spaced lines, inserting spaces and numbers, exactly as shown in the diagram, when the paper has been suitably folded to enclose a slide carrying scales B, C and F. The distance between the 20 and the 25 scale division on scales A and C may be taken as the unit distance, when the space between the 5 and the 10 must be made 3 times as great, and so on. The necessity for using any specially (*e.g.* logarithmically) ruled paper has been avoided, and all the distances concerned are equal to, or very simple multiples of, the unit distance. In order to accommodate the two new scales F and G, scale D has been moved to the upper part of the rule, and scale E has been placed on the lower line. In addition, upon scale C a long line has been drawn at the 50 mark, which ends in an arrow-head, pointing on scale E to the dilution which is being employed in a particular case. Reference to Part I. will make all this part of the arrangement clear. The graduations on scale F represent the ratio between the strengths of the strong solution to be determined and that of the weaker standard solution, and those on scale G show the final burette reading required.

Before describing how the scale is used in practice, it may be well first to show the simple mathematical operations which it has been constructed to perform.

Let A represent the volume of strong liquid taken (c.c.); f, the strength of strong liquid (grm.-equivalents per c.c.); B, the volume to which A is diluted; C, the volume of diluted solution measured out; E, the number of times the volume is increased by dilution; F, the ratio of strengths of strong liquid and weaker standard; G, the final burette reading, *i.e.* volume of the weaker standard needed for each aliquot part; and g, the strength of weaker standard solution.

Then we have at once by definition,

$$E = B/A, \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots$$

$$\text{and } F = f/g. \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \text{ii.}$$

Also, the strength of the diluted solution is given by $fA/B$, so that we have

$$CfA/B = Gg.$$

Therefore $AC/B = Gg/f = G/F$, by ii.

Let D be such that $AC = BD$ ..... iii.

where, by i, $B = AE$ ..... iv.

and $G = FD$ ..... v.

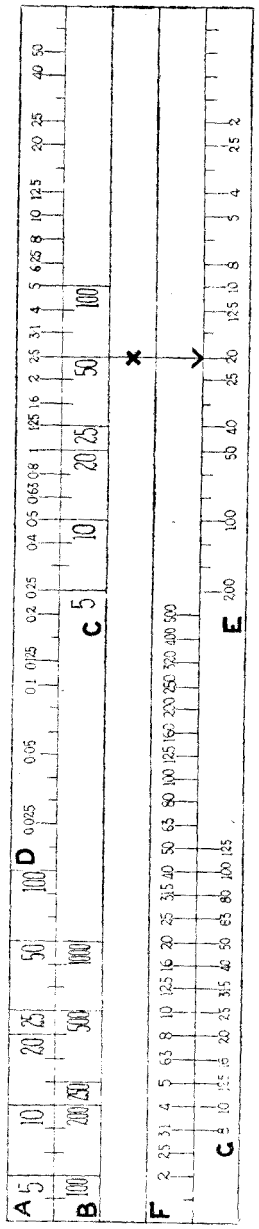
Scale I. was constructed so that iii. and iv. hold good, and D is obtained from v. by preliminary calculation.

In Scale II., equation v. is satisfied in addition to the other two. It may be noted here, in passing, that since equation iii. is symmetrical in A and C, evidently the operation of taking volume A, making it up to volume B, and abstracting volume C, will have the same effect (*i.e.* will give the same final burette reading) as if volume C is taken, made up to B, and A is abstracted. This rule, if remembered, may prove useful in certain cases.

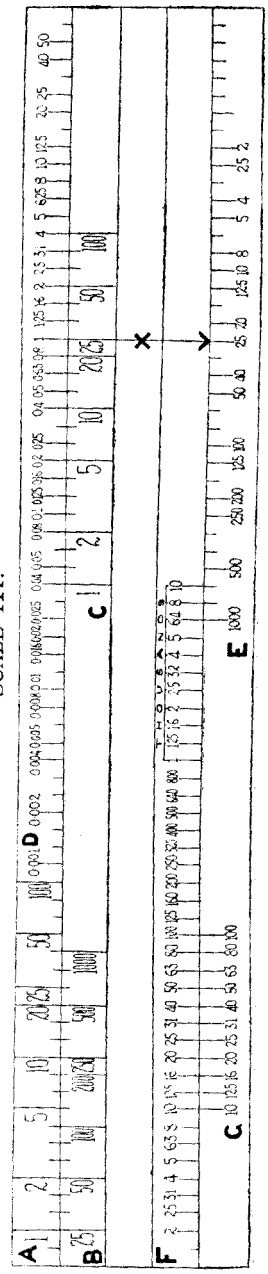
It is hoped that the use of Scale II. can now be made clear. It can be used, like Scale I., either directly or indirectly. For direct use, let us suppose we decide to take 10 c.c. of a strong solution, dilute to 500 c.c., and take 50 c.c., and that we know the strong solution to be approximately 40 times as strong as the weaker standard. (If this latter ratio is not already approximately known, it must be determined by a rough titration.) The question is: How many c.c. of standard solution will be needed for each aliquot part? The answer is, G=40 c.c. For, evidently we have A=10, B=500, C=50, and F=40; so placing the 10 on scale A opposite the 500 in scale B, we read off 40 on scale C opposite 40 on scale F. If C is not equal to 50 (*i.e.* where the long line occurs on the slide), a second move of the slide is necessary, as follows. Suppose we make up A=25 c.c. to B=200 c.c., and take out C=10 c.c., and the ratio F=20; then we place the 25 on scale A opposite the 200 on scale B, and read off scale D opposite the 10 on scale C. This gives 1·25, and the long line at the 50 mark on scale C must be brought to this point on scale D before the reading is taken on scale G. When this is done, scale G reads 25 opposite the 20 on scale F, so that the required number of c.c. =25. This method can be applied quite generally.

With regard to the indirect method of use, the problem is: What dilution shall we employ to obtain a given final burette reading? This is obtained as follows. As in the above example, unless the strength of the stronger solution be already known approximately, there must first be a rough titration. This gives F, the ratio defined by equation ii. This value of F on scale F is brought opposite to whatever burette reading is considered convenient on scale G. The upper end of the line at the 50 mark on scale C will now show the value D on scale D. This number D measures the effect of the operation of dilution and abstraction of a measured part. For instance, suppose the ratio of the strengths of the two solutions is approximately 10, and 25 c.c. is considered a convenient burette reading, bringing the 10 on scale F opposite the 25 on scale G, the long line on scale C then reads 2·5 on scale D in accordance with equation v. The scale is now correctly set for measuring out 50 c.c. of diluted solution, and if this is a convenient amount, no further change need probably be made. It will then be seen at once by examining coinciding numbers on scales A and B, that if 5 c.c. are made up to 100 c.c., or 10 c.c. to 200 c.c., and so on, taking out 50 c.c. as aliquot part, this will give the burette reading required. Which dilution is made will depend upon circumstances, *e.g.* upon which calibrated pipettes and measuring flasks are at hand, and how much of the strong solution is available for the purpose.

SCALE II.




SCALE III.



Evidently each dilution produces the same dilution ratio E, which is 20 in this case, as is shown by the arrow at the bottom of the long line on scale C. Now suppose that a quantity of 50 c.c. is regarded as an inconvenient amount of diluted solution to take, and that, for instance, 25 c.c. is preferred, it is only necessary to bring the 25 on scale C opposite the number previously found on scale D, *i.e.* 2·5, when it will be found that 10 c.c. diluted to 100 c.c., or 20 c.c. diluted to 200 c.c., and so on, taking out 25 c.c., will give the same final burette reading, as the previous case. Here, however, the dilution is only 10 times, and this is indicated by the arrow on scale E. Note that the lower end of the long line, *i.e.* by the arrowhead, is always read on scale E, but the only purpose of the upper end of the line is to find the appropriate number on scale D so that scale C is always read opposite this number when once found for a particular case. The rule for use, therefore, is to fix the mind on the value on scale D which corresponds to the coincidence of the appropriate (or nearest) values on scales F and G, setting any volume on scale C to coincide with it, when scales A and B at once indicate how the liquid can be diluted, and what choice (if any) exists. Generally speaking, if 50 c.c. is a convenient amount of the diluted solution for removal at a time, then only one move of the slide is necessary; if not, then two moves are needed. Of course, it may so happen that, although 50 c.c. would be a convenient volume on scale C, a second move of the slide is necessary, if the corresponding volumes on either scales A or B or both are unsuitable. In any case, by placing different volumes on scale C upon the point found on scale D, all the possible variations will be included, and a suitable one can be selected.

This explanation is unavoidably rather lengthy, but in use, the operation is very simple, and will give in a couple of seconds what is required.

Exactly as in Scale I., it is only when the arrow indicates a number on scale E that any useful numbers on scales A and B coincide; hence this must always be arranged to happen before any readings are taken. For instance, if the ratio F is 8, and the number G required is 25, and these values on scales F and G respectively are made to coincide by means of the slide, scale D reads 3·1 opposite the long line, but the arrow is opposite a space on scale E, and therefore no numbers on scales A and B coincide. This does not, however, entail that this is an impossible case, for such a case only occurs when on first setting the instrument the upper end of the long line coincides with a space on scale D. This has not happened; therefore one must bring other values on scale C to the 3·1 mark on scale D till the arrow indicates a number on scale E. It will be found that when the 25 on scale C is placed opposite the 3·1 on scale D, the arrow indicates 8 on scale E, so that the desired result can be achieved by taking 25 c.c. of solution, diluting it to 200 c.c., and taking out 25 c.c. for each aliquot part. It so happens in this case that this is the only way in which this particular result can be achieved, and the other values on scale D which can only be reached in one way are the 50, 40, 1·6, and 0·025. In all other cases, it is possible to choose between two or more ways of continuation. There are 19 choices when D reads unity, and other values have intermediate numbers of choices. Only when the adjustment on scales F and G

brings the top end of the long line to a space on scale D is there no solution. In this case, it will be generally possible to perform an operation by shifting the value on scale G up or down to the next graduation. It will now be clear that when scale F and G are set, the upper end of the long line must be opposite a number on scale D, and when this is so, there may be anything from no choice to 19 choices as to how the operation may be performed.

Scale III. is a still further expanded form of Scale II., in which the 1 and 2 c.c. pipettes have been included on scales A and C, and the 25 and 50 c.c. measuring flasks on scale B. Here the arrow line has been placed at the 25 c.c. mark on scale C, as occupying a more central position. With this difference, this rule is used exactly as Scale II. Values of F up to 10,000 have been inserted, though this is considerably above what will generally be needed. In Scale III. the only values on scale D giving no choice as to procedure are 50, 40, 3·1, 0·016, and 0·001. When D reads 1, there are 31 possible cases, and other readings have intermediate numbers of choices, which will be found when the sliding scale is examined in detail.

With regard to the exactness of these scales, equations iii. and iv. are always exactly satisfied. The fitting in of equation v. on Scales II. and III. has given rise to certain approximations. Thus 31 on scale F opposite 63 on scale G will be found to give 2 on scale D, though the division yields a small remainder. The agreement is however sufficiently exact in practice. The problem to the solution of which the Scale is applied is evidently not to bring the final burette reading to an exact point, but only into a desired neighbourhood. In any case, the value F is not known with exactitude, so some approximation in the quotient G/F is inevitable. Hence, there is no disadvantage involved. If complete exactness for any reason is desired, Scales II. and III. can be used without reference to scales F and G, scale D being used as described in Part I., but the cases in which perfect exactness is not attained are relatively very few.

Three Scales have in all been described, though all are based upon the same principles, as set forth in equations iii., iv. and v. Scale I. has the advantage of simplicity of construction, though needing a short calculation before use in a given case. Scale II. includes all that is in Scale I., and avoids the preliminary calculation. Scale III. is the most comprehensive of all, and once it is set up, it possesses all the advantages of the other two Scales, with greater range of application. Every case that would normally come under consideration is included in it.

In conclusion, it may be of interest to notice the number of distinct cases which have been combined together in these Sliding Scales. In counting the number of "operations," this must be taken to mean the number of different ways in which a volume A can be made up to B and a volume C taken out, using the pipettes and flasks mentioned. This number is 169 in Scales I. and II., and 366 in Scale III.; further, when these are combined in all possible ways with various arrangements of scales F and G, the total number of combinations is 1,968 on Scale II., and 3,836 on Scale III.

# Note.

*The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.*

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## BOERHAAVIA REPENS (PUNARNAVA).

By Jitendra Nath Rakshit.

The valuable plant *Boerhaavia repens*, Linn. (Syn: *B. diffusa*), which grows in almost every part of India, is gradually proving itself to be a very valuable remedy for dropsy. Some work has already been done upon it by Ghosal and McKay (*Biochem. J.*, III., Nos. 1 and 2) and by Basu (*Indian Med. Gazette*, 1910, 132), but mainly in connection with its medicinal properties. The author also confirms its curative properties in very complicated cases of dropsy, as observed among his own patients. The drug is gradually becoming an article of trade, and hence it is desirable to have its general analysis recorded.

For this purpose plants spreading to about 2 feet diameter, on the ground, and having roots 8 to 12 inches long and ½ to 1 inch in diameter, and stems of a reddish tinge were used. The roots taken from a bag of the freshly collected drug containing entire parts of the plants weighed 3180 grms., and the stems with leaves 1320 grms. These were dried on a steam-heated table, powdered, sieved through cloth and analysed. Estimations of starch were made by the hydrochloric acid method as adopted by the A.O.A.C. and described in *Allen's Commercial Organic Analysis* (Vol. I., 1909, p. 420). The amounts of extract were estimated by heating 2 grms. of the powder in an Erlenmeyer flask with 100 c.c. of the solvent on a steam bath, shaking the flask at intervals for four hours while keeping the mouth loosely closed with a cork, and leaving it overnight, and filtering the extract the next morning. Ten c.c. of the filtrate were then evaporated on the steam bath and the residue dried in the steam oven until constant in weight.

The following results were obtained:

| | Moisture | | Dry Sample | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Fresh sample | Air-dried sample | Ash | Water-Sol.-matter | Sol. in 1% Ammonia | Sol. in HCl. | Sol. in 90% Alcohol | Sol. Carbohydrates before inversion (as invert sugar) | Total Sol. Carbohydrates after inversion (as invert sugar) | Insol. Carbohydrates as starch |
| Roots, per cent. | 60·0 | 11·5 | 10·5 | 31·0 | 23·0 | 44·0 | 9·0 | 1·6 | 4·1 | 35·6 |
| Stems & leaves, per cent. | 70·5 | 13·0 | 18·9 | 11·0 | 11·5 | 27·0 | 11·0 | 1·4 | 3·2 | 17·2 |

Dilute hydrochloric acid extracts of all parts of the plant, after treatment with lead acetate and removal of excess of lead by means of dilute sulphuric acid, gave the usual alkaloidal precipitates with Mayer's reagent, iodine solution, picric acid and phosphomolybdic acid. The aqueous extracts were neutral to litmus, and gave the usual starch reaction with iodine, but no alkaloidal precipitate with Mayer's reagent after their acidification with dilute acids. When hydrochloric acid extracts of equal strength were treated with Mayer's reagent in equal quantities, the extract from stems and leaves often gave a larger volume of alkaloidal precipitate than that from the roots.

Ghazipur, U.P., India.

# Notes from the Reports of Public Analysts.

*The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.*

## CITY OF BIRMINGHAM.

REPORT OF THE CITY ANALYST FOR THE FOURTH QUARTER, 1922.

DURING the quarter 1127 samples were submitted, 979 were analysed under the Sale of Food and Drugs Acts, and 139 samples were analysed for various Corporation departments.

Of the 979 samples examined under the Food and Drugs Acts, 886 were bought informally, of which 23 were adulterated. Ninety-three samples were then bought under the provisions of the Acts, and of these 27 were adulterated.

MILK.—Of the 465 samples examined, 30 contained less than 11·5 per cent. of total solids. The percentage adulterated was 6·5. The average composition of all the samples examined was 3·92 per cent. of fat and 8·74 per cent. of solids-not-fat.

BUTTER.—Six of 63 samples contained water in excess of 16 per cent.

CREAM.—Two samples contained 0·3 and 0·2 per cent. of boric acid, respectively, without a declaratory label, and each vendor was fined £1 under the Sale of Milk and Cream Regulations.

SPONGE CAKES.—Samples were taken from four vendors who had sold informal samples in 1921. Two contained 35 grains and 23 grains of boric acid per lb., and each vendor was cautioned. The third sample contained 33 grains of boric acid, and the vendor was fined £10 for selling an article injurious to health. He gave notice of appeal, but did not proceed with the appeal. The fourth sample, a jam sandwich, contained 28 grains of boric acid per lb.

PORK SAUSAGE, POLONY.—Three informal samples contained 37, 35 and 33 grains of boric acid per lb. respectively. Another sample was unadulterated.

MUSTARD.—Ten samples were genuine, but one contained 35 per cent. of wheat flour. A further formal sample was unobtainable.

TREACLE.—The single sample was adulterated with about 20 per cent. of glucose.

EUCALYPTUS OIL.—Eleven samples were of satisfactory quality. The sp. gr. ranged from 0·915 to 0·927, the rotation in 100 mm. tube from +0·5 to +2·1, the butyro-refractometer readings from 51 to 55, and in each case more than 55 per cent. of cineol was present.

J. F. LIVERSEEGE.

## Dominion Laboratory, New Zealand.

FIFTY-FIFTH ANNUAL REPORT, 1921.

THE total number of samples examined during the year 1921 was 4804, of which 3321 were for the Department of Health. The remainder were analysed for the different public departments, including 387 for the Customs, 248 for the Explosives Branch, 504 for the Mines Department, and 29 for the Justice Department (Police).

Seventy-seven samples of water from mineral springs, for drinking purposes or for use in boilers, were examined.

DEPARTMENT OF HEALTH.—Of the samples taken under the Food and Drugs Acts, 2506 were milks, of which 1724 were samples taken in Wellington, 14 of these being adulterated. Of the samples from the country district, 70 were adulterated or below the standard.

Two baking powders were below strength and one contained a large amount of alum. Thirteen samples of ice-cream were deficient in butter fat, and six had been thickened with cream. Salicylic acid was found in 24 lemonades, and half of the samples of lemonade from all parts of the Dominion contained saccharin, in contravention of the regulations.

JUSTICE DEPARTMENT.—The samples examined included liquors, drugs, samples relating to cases of suspected incendiarism, and exhibits for examination for poisons. In one case crude carbolic acid, and in another potassium cyanide, was found. Carbolic acid was also present in another case, but experimental and other evidence indicated the possibility of its having been produced by bacterial action in the body (*cf.* ANALYST, 1922, **47**, 294).

DEPARTMENT OF MINES.—Samples of coals, oil shales, limestones, clays, ochres, and rocks were analysed for the Geological Survey; ores of copper, iron and mercury for the Head Office; whilst prospectors' samples included several varieties of useful clay, manganese and copper ores, quartz containing gold in payable amounts from several districts, and beach sand which contained a few grains of gold per ton.

RESEARCH WORK.—Further work on clays has shown that New Zealand possesses a variety of useful clays. The few samples of native ochre examined compared favourably with imported ochre for colour and tinting power.

LIGNITE.—The yield of oil from New Zealand lignites would be insufficient to repay the cost of the process of low-temperature distillation unless the residues could be rendered marketable, *e.g.* by briquetting.

FUSAIN.—Samples of very fine coal resembling the fusain from British coals were collected from mines in which there is a tendency to spontaneous ignition of the coal. These samples were of different composition from British fusain.

J. S. MACLAURIN.

# Legal Notes.

*Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.*

## CATTLE FOOD CONTAINING CASTOR BEANS.

*Pinnock Brothers* v. *Lewis and Peat, Ltd.*

THIS case, which was heard in the King's Bench Division on February 7, 1923, was a claim for damages for breach of contract for the sale of cattle food.

The plaintiffs had bought, by a contract in writing, from the defendants, one hundred bags of East African copra, and had re-sold them after delivery to another firm, who, in turn, had sold them to a third firm, who had manufactured the copra into cattle cake and re-sold it. All the cattle to which the cake or meal had been

supplied became seriously ill, and it was found, on analysis, that castor beans had been mixed with the copra cake.

Mr. Bevan, for the plaintiffs, submitted that the clause in the contract providing that the goods were not free from latent defects, not apparent on reasonable examination, did not apply to a poisonous substance in the copra cake. In any case, the clause was a mere exclusion of a warranty and did not affect the condition of the contract that the food sold should be of the contract description. The plaintiffs would also rely on Sec. 1, Subsecs. (3) and (4) and Sec. 6 of the Fertilisers and Foodstuffs Act, 1906.

Mr. Jowitt, for the defendants, submitted that the action must fail on several grounds. In the first place the arbitration clause in the contract prevented the action from being brought after the lapse of fourteen days. Secondly, the vendors were not liable for latent defects, and in this case the amount of castor bean in the cake was very small, and the article delivered was commercially the article required by the contract. And thirdly, the damages were too remote, for the cake had passed through the hands of a string of buyers, and the defendants could not be liable to buyers far down the string.

Evidence was called for the defence to prove that if a buyer wished to make sure that copra cake did not contain castor bean he should stipulate to that effect in the contract. In fact, all contracts made on the Liverpool market included a special warranty of freedom from castor bean.

Mr. Justice Roche, in delivering judgment, said that the presence of an arbitration clause in a contract was no bar to an action unless it expressly provided so. In this case the arbitrator refused to go into the merits of the case because notice had not been given in time. In his view the word "defect" could not cover the case where the article supplied was not the article contracted for, but was something entirely different. Copra cake *plus* castor beans in the proportions to be found in this case could not properly be described as copra cake at all. That finding made it unnecessary to decide whether, if there was a defect here, it was latent, but he thought the condition of the cake was not latent in that it could have been discovered by the exercise of reasonable care. He found as a fact that it was within the contemplation of the parties that the cake would be used for feeding cattle and for nothing else. The mischief was discovered, and was communicated to the defendants as soon as possible. Then it was said that there was a chain of buyers, and the defect ought to have been discovered by the plaintiffs by the exercise of reasonable care. With regard to this he found as a fact that except to an expert the condition of the cake was not apparent, and in dealing with such a small quantity of goods it was not negligent or unreasonable of the plaintiffs to omit to have an analysis made. The cost of analysis would have swallowed up the profit on the transaction, and the omission to have it made did not break the chain. If it was the duty of anyone to have goods of this kind coming from abroad analysed, he thought it must be the first person who put the goods on the market. The plaintiffs were entitled to rely on their vendors to supply the article contracted for, and they were further protected by the Fertilisers Act, 1906. There must be judgment for the plaintiffs for the amount agreed (£550) and costs.

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## CAMPHORATED OIL SUBSTITUTE.

On February 22, 1923, a J. Blyth, a pharmacist, was charged at the Aberdeen Sheriff's Court with selling camphorated oil containing less than 20 per cent. of camphor.

The defendant pleaded guilty, and stated that an assistant had sold "Compound Camphorated Oil," which, although not officially recognised in the Pharmacopœia, was a perfectly legitimate liniment containing 2 per cent. of camphor. The assistant had explained this at the time of the sale, and the quantity sold on that day was labelled "Compound." There was no question of putting money in his pocket. In his opinion, the compound liniment was quite as efficacious and was pretty much the same type as camphorated oil.

The Procurator-Fiscal pointed out that people had been asking for camphorated oil and had received the preparation made by the accused. He did not think that the addition of the word "compound" would convey much to the ordinary customer. Accused had said that the liniment was the result of war-time conditions, but he understood that camphorated oil was now back to pre-war prices. Samples of camphorated oil had been taken from all the chemists in Aberdeen, and accused seemed to be the only one in the trade who was selling such a preparation.

The Sheriff, in imposing a fine of £3, said he was unable to accept the explanation of the accused as satisfactory. It was necessary for him to give the public what they asked for, or to tell them what they were getting, instead of trying to give them something they did not really want.

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# The Cleaning and Restoration of Museum Exhibits.

## SECOND REPORT UPON INVESTIGATIONS CONDUCTED AT THE BRITISH MUSEUM.*

PRINTS AND PICTURES.—In the first report a method of restoring white lead pigments on drawings was described in which the vapour of hydrogen peroxide is brought into contact with the discoloured surface (ANALYST, 1922, **47**, 122). The same method is also effective for removing the foxed markings on engravings, although a longer treatment is required for this purpose and there is some risk of the reagent causing disintegration of some kinds of paper, apparently owing to its oxidising action upon the sizing. Where an ink with an iron basis has been used for a drawing, hydrogen peroxide will change it to a faint yellow, although the colour can be restored more or less completely by treating the bleached place with potassium ferrocyanide and ammonia. Hence, before treating drawings supposed to be done in sepia, it is necessary to apply the test to a small portion of the surface. Sepia appears not to be affected by hydrogen peroxide vapour. Information as to whether a drawing has been done with sepia or bistre has been obtained in many instances by the use of sodium hypobromite solution, a minute drop of which applied to the pigment gives a red coloration with sepia before bleaching it, whereas in the case of bistre there is no red coloration and the bleaching is imperfect, so that small particles, visible with a lens, remain distributed over the treated surface.

A useful reagent for removing red stains such as those produced by the dyes used for red ink is a solution of sodium hydrosulphite ("Hydros"), prior to the application of the bleaching agents described in the former report.

Under certain conditions thick paper or mounts become wrinkled when exposed to the moist atmosphere of hydrogen peroxide vapour, and in such cases

* Department of Scientific and Industrial Research. H.M. Stationery Office. 1923. Price 2s. net.

it is best to treat the discoloured pigment with a solution of hydrogen peroxide in ether, which is applied by means of a camel's hair brush.

Sometimes a pigment is not only discoloured but is also inclined to become detached from the paper. Such flaking is prevented by coating the surface of the drawing with an acetone solution of cellulose acetate, any undesirable gloss being subsequently removed by treatment with acetone. Cellulose acetate varnish has also been successfully used as a preservative coating for disintegrating mural tablets and as an adhesive for attaching fragments of paintings on silk to a base of cloth.

STONE AND EARTHENWARE.—The disintegration of objects of stone and earthenware has been found to be due to alternate solution and crystallisation of soluble hygroscopic salts (chloride, sulphate, nitrates and ammonium salts) derived from an outside source. The remedy is to extract these soluble salts by repeatedly soaking the object in water.

SILVER.—The method of removing incrustations from silver objects by treatment with formic acid has continued to give good results, and the use of zinc dust in association with very dilute sulphuric acid has proved a useful reagent in obstinate cases.

LEAD.—After treatment with solutions such as those recommended in the last report an additional protection has, in some instances, been given by covering the object with a thin film of varnish prepared by dissolving about 5 parts of celluloid in 100 parts of a mixture in equal proportions of acetone and amyl acetate. Devices on the surface of objects made of lead are frequently distorted owing to the expansion of the metal. This has been found to be caused by the fact that internal corrosion, probably centring about an organic nucleus, is more vigorous than the external corrosion.

COPPER AND COPPER ALLOYS.—In some cases the acid reagents previously described cannot be used, and an alkaline solution is more suitable. A suitable reagent of this type is a solution of 3 parts of Rochelle salt and 1 part of sodium hydroxide in 20 parts of water. Some objects can also be successfully cleaned by treatment with granulated zinc and dilute (5 to 10 per cent.) sodium hydroxide solution.

WOODEN OBJECTS.—As in the case of stoneware, disintegration of objects of wood is frequently due to the crystallisation of soluble hygroscopic salts. These may be removed by extraction with dilute (5 per cent.) acetic acid, the excess of which should then be removed by thorough washing. The object is next immersed in 0·5 per cent. mercuric chloride solution, and is finally coated with celluloid varnish, as described above. For the destruction of insect life the most effective agents are carbon disulphide and hydrogen cyanide in the form of vapour, but, contrary to expectation, carbon monoxide is not fatal to *Anobium punctatum*. The objection to the use of chloroform and tetrachlorethane is that chlorine may subsequently be liberated and have an injurious effect upon any coloured pigment present. Carbon disulphide vapour does not appear to have any injurious effect upon oil paintings containing flake white (white lead), for in experiments in which holes were bored into the back of such a painting on wood almost to the level of the film of pigment, and repeatedly filled with liquid carbon disulphide, not the slightest discoloration of the lead paint was observed.

# Institute of Chemistry.

At the 45th annual general meeting of the Institute of Chemistry, held at 30, Russell Square, on 1st March, the President, Mr. A. Chaston Chapman, F.I.C., F.R.S., in presenting the report of the Council, remarked on the progress of the organisation of the profession of chemistry under the Institute. The roll of Fellows and Associates had increased during the past twelve months by 421, to a total of 4062, and the register of students, by 72, to a total of 955.

The Institute was taking an increasing part in the affairs of the country, and was frequently appealed to by Government Departments and other authorities for advice and help. Natural science—and in this connection chemistry must be given a position of great prominence—was by far the most important dynamic factor in human progress. Notwithstanding its liability to abuse, its discoveries had on the balance made enormously for the greater good and greater happiness of the human race. Science was coming into its own and scientific men would be given their proper status and rightful place in the affairs of the country. The Institute, in common with other societies and associations, was actively promoting a spirit of solidarity and comradeship among all who were concerned with chemistry, with a view to welding the profession into one coherent whole. With the advances which chemistry was making in all directions and with the high degree of specialisation to which they were tending, many societies had come into existence, but the dangers of decentralisation were greatly lessened by the practice of holding joint meetings of the societies.

The direct utilisation by the State of the services of the professional chemist was a matter not only of immediate concern to chemists themselves, but was of high importance to the community at large, and in the interests of the country no less than in those of its members the Institute was bound to do all it could to ensure that the relations between the appointing authorities and those who held official chemical positions, were of a satisfactory character. Unfortunately, some public bodies did not appear to be aware of the lengthy and expensive nature of the chemist's training, or of the difficulties and responsibilities connected with his work, and consequently the Institute found, from time to time, that the advertised conditions of certain public bodies were not commensurate with the importance of the services demanded. The mere saving of money was not always identical with true economy, and it was clearly of public importance that men of good general education, of high professional attainments, and of high moral character, should be chosen to fill these public positions, while it was obvious that appointing authorities would seriously limit the field for selection unless the conditions offered were such that men of the right class would be willing to accept. For the appointment of public analyst the remuneration offered was often entirely inadequate, and, in some cases, even below that paid before the war, notwithstanding the enormously increased work and responsibility devolving on the shoulders of the public analyst. A statement on the whole subject would shortly be published by the Institute jointly with the Society of Public Analysts. There was a tendency on the part of local authorities to utilise the services of unqualified or imperfectly trained persons for carrying out what were regarded as simply routine processes, a practice against which the Council had felt bound to protest vigorously, on the ground that it constituted a serious danger to the community and involved a waste of public money. Even the simplest process required some skill and experience if pitfalls were to be avoided and mistakes obviated, which might, in some cases, have serious consequences.

The Institute had also been obliged to complain of the competition of state-aided institutions with private practitioners, and it was a source of gratification that the Ministry of Agriculture and Fisheries had recognised the legitimate grievance of the private practitioner and had taken steps towards limiting the agricultural work done in the Institutions receiving its grants to that required for strictly educational purposes or necessary to the advisory work of the Institutions.

The disinterested zeal of the scientific worker was something without parallel in the whole world, but it was not wise for any country to presume too much on this disinterestedness. Science was one of the greatest and freest of all givers, but it had a right to demand that it should receive that recognition and that proper position in the councils of the country to which it was entitled. The indirect effect of proper State treatment was very great. The rulers of Germany knew very well how much science could do to increase the greatness of their country in times of peace and its chance of success in the event of war. Germany had not changed. A leading German industrial chemist had said lately that notwithstanding Germany's position of virtual bankruptcy, the State, at the instigation of the commercial committee of the Reichstag, had come to the help of the great chemical and physical societies, particularly to that of the Kaiser Wilhelm Institute, and if the state could not continue financial aid the German people themselves must give their last mark to maintain science.

Referring to the prospects of the profession, the President said chemistry had great attractions for most boys, and there were many induced by a liking begotten in the school laboratory to embark upon a profession for which they had perhaps no real aptitude. A chemical career was not a succession of fascinating experiments, but it involved a good deal of hard work of a comparatively unattractive character, made very great demands on its devotees, and called for much self-sacrifice on the part of those who adopted it as a profession. He advised parents who consulted him on the matter to put their sons or daughters to some other calling unless they had so strong a love for the subject that the necessary hard work would lose its element of drudgery and the sacrifices would be cheerfully borne. Although the supply of qualified chemists exceeded, for the moment, the demand, he did not think that there was cause for serious alarm. The profession had attracted a larger number of young men during the last four years than in any previous corresponding period, but he was very optimistic in regard to the future of chemistry, and it was a remarkable fact that, notwithstanding the increased output from the colleges and the intense industrial and commercial depression, the new members of the profession were being steadily absorbed. This absorption might be taken as a definite indication that chemistry was more highly valued by the manufacturer than formerly, and as evidence that the leaders of industry and commerce were turning more and more to science to assist them in the solution of their various problems. He did not forget, however, that there was, for the time, a surplus, and he appealed to the members to do all they could to help those who were out of employment to secure appointments.

The report of the Council and financial statements were received and adopted, and the officers, council and censors for the ensuing year were elected.

The Meldola Medal was awarded, for the second time, to Dr. Christopher Kelk Ingold.

# Ministry of Health.

## Circular 381.

THE following Circular has been sent to the Clerks of Authorities administering the Food and Drugs Acts.

### I.—BORIC ACID IN CAKE.

I AM directed by the Minister of Health to state that he has received communications from a number of local authorities with regard to the presence of boric acid in cake, some samples (especially of sponge cake) having been found to contain a somewhat high proportion of this preservative.

In view of the fact that certain varieties of sponge cake are commonly used for the food of infants and invalids the excessive use of boric acid is particularly undesirable, and steps have been taken to ascertain the ingredient through which it has been introduced. It has been found that while such ingredients as butter or margarine may contain small percentages of boric acid, any large percentages found in cake are mainly due to the use of liquid whole egg, a material manufactured from imported egg yolks, preserved by means of boric acid, and dried egg albumen.

The matter has been under discussion with representatives of the Bakery Allied Traders' Association, which, it is understood, includes most of the firms manufacturing or trading in liquid whole egg, and that Association have agreed to take certain steps with a view to effecting a reduction in the amount of boric acid in liquid whole egg and in sponge cakes. The Association have passed two resolutions which are to be implemented by bonds signed by every individual member of the Association.

The undertaking given in pursuance of the first resolution was in the following terms:—

"I/We the undersigned, hereby undertake that on and after the 22nd February, 1922, that I/We will not manufacture nor import Liquid Whole Egg containing more than one per cent. of Boric Acid."

"I/We also undertake not to purchase after the 22nd February, 1922, new season's yolks for shipment, containing more than 1·5 per cent. of Boric Acid."

"It is understood that this does not interfere with stocks already on hand, manufactured or in course of manufacture, nor does it apply to contracts for shipment which have been already made and not completed."

A further resolution passed in July, 1922, was subsequently rescinded, but on the 1st February, 1923, the Association resolved that its members should sign a bond that in future the sale of liquid whole egg should be under the following conditions:—

"(1) Sale only to be made on the understanding that Liquid Whole Egg must not be used in the manufacture of Sponge Fingers, Sponge Biscuits and 1½d., 1d. and 2d. plain Sponge Cakes."

"(2) That all Invoices should bear a statement to the buyer that Liquid Egg must not be used in the manufacture of Sponge Fingers, Sponge Biscuits, and 1d., 1½d. and 2d. plain Sponge Cakes."

It may be observed that the fingers, biscuits and small cakes to which this resolution relates are those which are most likely to be used by infants and invalids.

The Minister is advised that if effect is given to the decisions of the Association the danger of the presence of excessive preservatives in cake will be substantially reduced. Local authorities will, of course, be able, as at present, to take proceedings under section 3 or section 6 of the Sale of Food and Drugs Act, 1875, in any case in which they consider that the amount of boric acid found in a sample of cake (whether within the scope of the Association's second resolution or not) is excessive.

### II.—IMPORTED PRODUCE.

The Minister desires to take this opportunity of correcting a misapprehension which has arisen in some quarters owing to the wording of paragraph 3 of Circular 360. The words "Imported Produce" should be substituted for "Imported Meat" in that paragraph, as the provisions of the Sale of Food Order, 1921, which remain in force until the 31st December, 1923, include clause 8 (relating to the labelling of imported eggs) as well as clause 7 (which in its present form relates only to meat). These provisions are set out in the Board of Trade's memorandum of September, 1922.

Further copies of this Circular may be obtained through any bookseller or directly from H.M. Stationery Office.

# ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

## Food and Drugs Analysis.

**Detection of Hypochlorites and Chloramines in Milk and Cream. P. Rupp.** (*Amer. J. Pharm.*, 1923, **95**, 49–50.)—Owing to the extended use of these chlorine compounds for disinfecting utensils in dairy work their intentional or accidental occurrence in milk is not unlikely. The detection of the available chlorine in the compounds when added to milk presents difficulties owing to the combination of the chlorine with the proteins. The following method has therefore been devised:—Five c.c. of the sample are shaken with 1·5 c.c. of a freshly prepared 7 per cent. solution of potassium iodide and the colour noted. If there is no perceptible change 4 c.c. of dilute hydrochloric acid (1:2) are added, the curd thoroughly stirred with a glass rod flattened at the end, the colour again noted, and the tube placed for 10 minutes in water maintained at 85° C., after which it is rapidly cooled in water, and the colour again noted. Finally, from 0·5 to 1 c.c. of starch solution (1 grm. in 100 c.c.) is added, and the colour reaction observed. Milk containing 1 part of available chlorine in 1000 acquires a distinct reddish tint, and, at a dilution of 1 in 2500, is still slightly coloured, as compared with a control sample. With 1 part of chlorine to 5000 of milk the mixture becomes pale yellow on the addition of the iodide solution. When heat is applied the delicacy of the reaction is increased, and 1 part in 50,000 can then be detected. Milk which has been kept for 48 hours in an ice box still gives the reaction, but, if it has been kept at the ordinary room temperature, the test is less sensitive. Milk pasteurised at 145° F. reacts like raw milk, as does also 20 per cent. cream. It is best to examine both the curd and the liquid below it.

**Grading of Flour by Enumeration of the Wheat Hairs present. G. L. Keenan.** (*Bull. U.S. Dept. Agric.*, No. 1130, 1923.) Owing to the difficulty encountered in the microscopical counting of bran particles the author has applied the same principle by enumerating the wheat hairs and hair fragments, which are more readily identified. Five mgrms. of the sample are weighed out, transferred to a micro slip ruled with horizontal lines 1 mm. apart, and well mixed with 4 drops of 50 per cent. chloral hydrate solution. A cover glass, 22 mm. square, is applied and the slide is gently warmed until the preparation is transparent and, after cooling, the number of hairs and portions of hair are counted (each being given a value of 1) under a magnification of 180 diameters. Since commercial flours show great variations in different mountings of the same sample a number of determinations should be made and the average value taken. The examination of a large number of samples, the results of which are recorded, showed that corresponding samples from different varieties of wheat gave closely agreeing values, but that the counts on "break" flours were much higher than on "middlings" flours. The

following counts were obtained on different grades:—Patent flours, 18 to 21; straight flours, 34 to 44; clear flours, 68 to 110; and low-grade flours, 109 to 182.

T. J. W.

**Separation of Meat Proteins. C. R. Moulton.** (*J. Assoc. Off. Agric. Chem.*, 1922, **6**, 76–85.)—Approximate separation of meat proteins was carried out by extracting the meat with cold water and diluting the mixed extract and washings to a convenient volume (*A.O.A.C. Methods*, 1920, 214). Separate portions of this solution were treated (*a*) with an equal volume of cold saturated zinc sulphate solution to precipitate globulin, (*b*) by neutralisation with excess of moist freshly precipitated magnesium carbonate, whilst the liquid was heated on a boiling water bath, by which means albumen and globulin were coagulated, and (*c*) by saturation with zinc sulphate and the addition of sufficient sulphuric acid to cause complete precipitation of the albumen, globulin and proteose. The amount of 50 per cent. sulphuric acid required per 100 c.c. of the extract was found to be for 7 mgrms. of coagulable nitrogen 1 c.c.; for 12 mgrms. 3·3 c.c.; and for 15 mgrms. about 5 c.c. Attempts were made to estimate the amino acid and "extractive" nitrogen by precipitation with tannic acid and sodium chloride, but great difficulty was experienced and erratic results were obtained. The nitrogen content of the various precipitates obtained was estimated by the Kjeldahl-Gunning-Arnold method. Detailed descriptions of the procedures adopted and several tables of the experimental results obtained are given. T. J. W.

**Colorimetric Estimation of Amino Acid Nitrogen in Foods by means of the "Ninhydrin" Method. H. Riffart.** (*Zeitsch. Untersuch. Nahr. Genussm.*, 1922, **44**, 225–239.)—The reagents required are:—(1) Potassium phosphate solution, 9·078 grms. per litre. (2) Sodium phosphate solution, 11·876 grms. per litre. (3) Neutral red solution, 0·1 grm. per litre of 50 per cent. alcohol. (4) 0·1 *N*, 0·01 *N*, and 0·0025 *N* sulphuric acid and sodium hydroxide solutions. (5) 1 per cent. "ninhydrin" solution (aqueous). (6) Comparison solutions, each containing 3, 6, 6, 10, etc., grms. per litre of asparagine (or of alanine, leucine, or other amino acid). Two c.c. each of the comparison solutions, of the solution under examination, and of a mixture of the phosphate solutions (2:3) are placed in separate test-tubes, 0·05 c.c. of neutral red solution is added to each, and, by the addition of acid or alkali, the colour of the solutions is made equal to that of the phosphate solution. In the case of the comparison solutions a few drops only of the most dilute alkali solution are required; if necessary, the stronger alkali or acid solution is used to neutralise the test solution in order to prevent great increase in volume. The neutralised solutions are treated with 2 c.c. of the mixed phosphate solutions and 0·5 c.c. of "ninhydrin" solution, placed in a boiling water bath for thirty minutes, or until the most dilute comparison solution is coloured blue-violet, then cooled, diluted to 100 c.c., and the colour of the test solution compared with those of the comparison solutions. Acid amides, creatinine, substances containing imido groups, aldehydes, ketones, lactic acid, xanthine, etc., do not interfere with the estimation, but if ammonia is present it must be removed from the amino acid

solution by vacuum distillation before the estimation is commenced. Proteins must also be removed from the amino acid solution, preferably by dialysis. Fresh beef contains 0·076 per cent. of amino acid nitrogen, veal 0·050 per cent., and horse flesh 0·036 per cent. Frozen (Argentine) meat contains about the same amount of amino acid nitrogen as does fresh meat. Incipient decomposition does not appear to be accompanied by a sudden rise in the amount of amino acid nitrogen. The quantity of the latter in fresh milk is 14 to 18 mgrms. per litre, and in boiled fresh milk 25 to 31 mgrms. per litre; when milk is kept, the quantity increases more rapidly in fresh milk than in boiled milk. W. P. S.

**Pilchard Oil. H. M. Langton.** (*J. Soc. Chem. Ind.*, 1923, **42**, 47–48T.)—Two samples of oil obtained from the fresh fish by a crude pressing process gave the following mean analytical figures:—Specific gravity, 15·5/15·5° C., 0·9320; saponification value, 187·7; iodine value (Wijs), 171·5; $n_D^{40}$, 1·4748; Reichert-Meissl value, 0·5; free fatty acids as oleic, 8·7 per cent.; unsaponifiable matter, 0·98, and insoluble fatty acids and unsaponifiable matter, 93·3 per cent.; viscosity (seconds Redwood at 40° C.), 118. The mixed fatty acids had the following characteristics: —Neutralisation value, 187·2 and 193·7; mean molecular weight, 291·1; iodine value (Wijs), 179·2; oxidised fatty acids, a trace; ether-insoluble bromides, 56·0 per cent.; melting point, 34·8° C.; and "titer," 28·2° C. On prolonged exposure to light the oil darkened somewhat and deposited resinous substances, whilst in the presence of air it gummed readily. It yielded good potash and soda soaps, which, however, developed fishy odours on keeping. D. G. H.

**Relationship between the Iodine Values and Refractive Indices of some Hardened Vegetable Oils. J. J. Sudborough, H. I. Watson and D. Y. Athawale.** (*J. Ind. Inst. Sci.*, 1922, **5**, 47–69; *J. Soc. Chem. Ind.*, 1923, **42**, 103–104A.)—Samples of the following oils were refined and hardened at 180° C.:—Cottonseed, linseed, groundnut, *Bassia latifolia*, sesame, sardine, castor, pongam, and coconut. The most efficient catalyst was found to be nickel-kieselguhr (25 per cent. nickel) prepared by direct reduction of a mixture of nickel carbonate and kieselguhr in hydrogen at 300° C.; this catalyst acted for about 44 hours at 180° C. without deteriorating. The products were then examined for iodine value (Winkler's method, *J. Soc. Chem. Ind.*, 1914, 872; 1920, 341A), and refractive index by means of the Abbé refractometer, and in the case of the first six oils the following equation was found to represent the relationship with an accuracy of about 0·0005:

$$n_D^{60} = 1{\cdot}4468 + 1{\cdot}03 \times 10^{-4}(\text{I.V.}) + 7{\cdot}3 \times 10^{-3}(\text{I.V.})^2,$$

the refractive index of the completely hardened oils approximating to 1·4468. With castor, and possibly pongam oils, the nature of the catalyst and time of hardening (factors not affecting the first six oils) caused a varying reduction of the hydroxyl groups, whilst the refractive indices of hardened coconut oils were much lower than those of other oils with similar iodine values. D. G. H.

**Estimation of Pectin in Fruit and Fruit Products. H. J. Wichmann.** (*J. Assoc. Off. Agric. Chem.*, 1922, **6**, 34–40.)—The sample is pulped, if necessary, thoroughly mixed, and 300 grms. are boiled with 800 c.c. of water for 1 hour, cooled, diluted to 2000 c.c. and filtered. Two hundred c.c. of the solution are evaporated to 25 c.c. and treated with 200 c.c. of 95 per cent. alcohol and, after the precipitate has subsided, it is filtered off and washed with 80 per cent. alcohol. The residue is completely dissolved in hot water, and the solution is evaporated to 25 c.c., cooled, treated with an equal volume of 0·8 per cent. sodium hydroxide solution, and allowed to stand 15 minutes. Forty c.c. of water are added, together with 10 c.c. of 10 per cent. hydrochloric acid, and the mixture is then boiled for 5 minutes. The precipitated pectic acid is removed by filtration, washed with hot water and transferred to a beaker, after which the treatment with sodium hydroxide and hydrochloric acid is repeated. The final product is washed into a platinum dish, evaporated to dryness, dried at 100° C. for 1 hour, weighed and ignited. The loss of weight represents the pectic acid. This method is considered to be more trustworthy than those involving the weight of pectin obtained after precipitation by alcohol, since that precipitate is liable to be contaminated with impurities. (*Cf.* ANALYST, 1922, **47**, 263.) T. J. W.

**The Acids Present in Certain Fruits. C. F. Muttelet.** (*Ann. Falsif.*, 1922, **15**, 453–455.)—The author has described previously (ANALYST, 1922, **47**, 398) a method for the estimation of malic and citric acid in fruits, depending on the isolation of the acids in the form of their barium salts and separation of these by fractional precipitation from dilute alcoholic solution, and he now records the results of estimations of the acids in various fruits. Apples, pears, cherries and quinces contain malic acid (0·3 to 1·3 per cent.), but no citric acid; gooseberries, currants, raspberries, and strawberries contain citric acid (1·0 to 3·5 per cent.), but no malic acid, whilst apricots and peaches contain both malic and citric acids. W. P. S.

**Examination of Authentic Grape Juices for Methyl Anthranilate. F. B. Power and V. K. Chesnut.** (*J. Agric. Research*, 1923, **23**, 47–53.)—The results are given of the examination of the juice from a large number of different varieties of grapes for methyl anthranilate. For the method used see *J. Amer. Chem. Soc.*, 1921, **43**, 377 (Abst. *J. Chem. Soc.*, 1921, ii., 557). All varieties of *Vitis labrusca* and nearly all the hybrids of *V. labrusca* contain methyl anthranilate, but the ester is not found in *Vitis vinifera*, the European grape, nor in *V. rotundifera* or *V. bourquiniana*. This ester, the detection of which may have diagnostic value in the determination of varieties, imparts a peculiar odour to grapes containing it, but does not completely represent their odorous constituents. Loganberry juice contains no methyl anthranilate. H. E. C.

**Tannin in Whiskey. R. W. Scott.** (*J. Amer. Pharm. Assoc.*, 1922, 1017; *Pharm. J.*, 1923, **110**, 134.)—It is frequently claimed that whiskey seized under the American prohibition laws has been in the possession of the defendant prior to

1919, and the proportion of tannin present is regarded as a valuable index of the age of the spirit. The method used is as follows:—One c.c. of the whiskey is diluted to 100 c.c. in a Nessler tube, and treated first with 1 c.c. of Folin's phenol reagent and then with 5 c.c. of a saturated solution of sodium carbonate, and the resulting blue coloration compared, after 10 minutes, with those given by standard solutions containing 0·0, 0·5, 1·0, 2·0, and 5 c.c. of standard tannin solution, prepared by dissolving 0·1 grm. of pure tannin in a litre of water. Folin's reagent is made by mixing 750 c.c. of water with 100 grms. of sodium tungstate and 20 grms. of phosphomolybdic acid (or 18 grms. of 85 per cent. molybdenum trioxide and 50 c.c. of 85 per cent. phosphoric acid), boiling the mixture for 2 hours beneath a reflux condenser, and then diluting it to a litre. Eleven authentic samples of American whiskey, bottled in bond, contained from 30 to 42·5 grms. of tannin per 100 litres, whilst a number of samples of "stretched" whiskey, which is ordinarily compounded from equal parts of alcohol, water and genuine whiskey, contained about 10·0 grms. per 100 litres, and this was also approximately the tannin content of a few samples of Canadian whiskey. While the test appears, in the case of American whiskeys, to be largely, if not exclusively, an estimation of tannin, it would probably include phenols in the case of Scotch whiskeys.

**Properties and Reactions of Strophanthin and of Ouabain. M. Tiffeneau.** (*Bull. Soc. Pharm.*, 1922, **29**, 68, 128, 184, 244, 249; *J. Pharm. Chim.*, 1923, **27**, 28–35.)—Strophanthin derived from *S. Kombe* consists of small aggregates of microscopic needles or micaceous lamellæ arranged in rosettes, whilst ouabain (from *S. gratus*) is readily obtained in rectangular plates by allowing a saturated aqueous solution to evaporate at the ordinary temperature. The $[a]_D$ of these compounds is $+29·16°$ and $-24°$ for the trihydrate and nonahydrate respectively. The solid substances may readily be distinguished by the following reactions:—Concentrated sulphuric acid yields with strophanthin a green colour, and with ouabain a rose red or yellowish brown, each of these reactions being sensitive to 0·001 mgrm. of the glucoside. With 80 per cent. sulphuric acid no coloration is given by ouabain, but strophanthin yields the same colour as with the concentrated acid. Strong sulphuric acid with various inorganic acids gives distinctive reactions, as for instance:—Phosphomolybdic acid yields a blue colour with strophanthin; tungstic acid gives a green coloration with strophanthin, but no coloration with ouabain; vanadic acid gives no colour with strophanthin, but an intense green with ouabain. Concentrated hydrochloric acid with phenol produces a violet colour with strophanthin, and the same acid with resorcinol reacts with strophanthin to produce a rose-red colour, but no reaction is given by ouabain with this reagent.

T. J. W.

**Analysis of Powdered Thyroid Gland and Detection of Adulterants. R. Fabre and H. Penau.** (*J. Pharm. Chim.*, 1923, **27**, 81–88.)—The pulverised preparation was examined as follows:—*Moisture*: About 1 grm. of extract was dried to constant weight at 105° C. (about 6 hours). The proportion of water varied, according to the preparation examined, from 6 to 10 per cent. *Iodine* was

estimated by weighing out 1·1 grms. into a nickel basin of about 63 mm. in height, 60 mm. external diameter, and 40 mm. internal diameter. The powder was mixed with 4 c.c. of alcohol; 5 c.c. of 20 per cent. potassium hydroxide solution were then added and, after 3 or 4 hours of contact and mixing, the whole was slowly brought to boiling point on a water bath, and boiling continued until a syrupy transparent mass remained. This was then carefully incinerated over an alcohol flame, and as soon as fumes ceased to be liberated the temperature was raised and the flame adjusted so that the operation was complete in about 30 minutes. The residue was taken up with a few c.c. of water, evaporated to dryness and ignited as before. On cooling, it was dissolved in a 0·2 per cent. solution of sodium chloride, and the small particles of carbon carefully broken up. After filtration, the remaining carbon was again treated with boiling sodium chloride solution, and the treatment continued until extraction was complete. Ten c.c. of 2 per cent. permanganate solution were then added to the clear and colourless filtrate, the whole boiled for 10 minutes to complete the formation of iodate and the excess of permanganate destroyed, while boiling, by the addition of a few c.c. of 95 per cent. alcohol. After cooling, the volume was made up to 220 c.c., the solution filtered, 10 c.c. of pure acetic acid and 1 grm. of ammonium chloride added to 200 c.c. of the filtrate, and the whole boiled for 10 minutes to destroy nitrites. After cooling, 10 c.c. of acetic acid and 1 grm. of potassium iodide were added to the clear liquid, and, after 5 minutes, the iodine was titrated with *N*/100 sodium thiosulphate solution, and the percentage of iodine calculated. A blank estimation was also made to ascertain the purity of the reagents. Products containing iodine, such as iodised albumins, which may have been fraudulently added to thyroid preparations, can be detected as follows:— One c.c. of pure ammonia solution and 9 c.c. of 95 per cent. alcohol are added to 0·5 grm. of the sample, the whole mixed for 15 minutes and filtered, the clear filtrate evaporated on a water bath, and the residue taken up with water and filtered through a wet filter paper in order to get rid of any fat. A few drops of ferric chloride solution and chloroform are added; a violet coloration indicates the presence of iodine, whilst with normal pure thyroid extracts no colour is produced.

D. G. H.

**Sensitiveness of some Cyanide Reactions. J. B. Ekeley and I. C. Macy.** (*Proc. Colorado Sci. Soc.*, 1919, **11**, 269–274; *J. Soc. Chem. Ind.*, 1923, **42**, 123A.)—Hydrocyanic acid may be detected in the following minimum quantities:— By the Prussian blue test, 1:170,000, or if applied to the distillate obtained after acidifying the solution with tartaric acid, 1:1,700,000; by the silver nitrate hanging-drop test, 1:19,000,000; and by the Schönbein guaiacum test, 1:55,000,000. In this last test chlorine, bromine, hydrogen peroxide and hydrochloric acid do not give reactions in greater dilutions than 1:1,000,000. D. G. H.

**Reaction of Neo-Salvarsan. K. Scheringa.** (*Pharm. Weekblad.*, 1923, **60**, 248.)—One drop of a 0·1 per cent. solution of neo-salvarsan gives a distinct violet coloration with a concentrated solution of ammonium persulphate. The

reaction is not specific, however, for a violet coloration is slowly produced by salvarsan. Aniline and *a*-naphthylamine give blue colorations with the reagent, brucine a red coloration, and diphenylamine a green coloration.

## Toxicological and Forensic.

**Rhododendron Poisoning. S. W. Hardikar.** (*J. Pharmacol. Exper. Therap.*, 1922, **20,** 17; *Pharm. J.*, 1923, **110,** 147.)—Cases of poisoning among sheep have been caused by the leaves of the rhododendron. The active principle is andromedotoxin, a non-nitrogenous compound, which may act either directly upon the heart, or by its narcotic action upon the higher centres of the brain. Part of the poison when injected hypodermically passes through the kidneys unchanged.

**Case of Mass Poisoning by Zinc.** (*Lancet*, 1923, **214,** 242.)—At a large institution near London over 200 persons developed typical symptoms of zinc poisoning immediately after a meal consisting of bread, margarine, stewed apples and tea. Subsequent investigation showed that the apples had been cooked in galvanised iron baskets placed in iron steamers, and that the fruit acids had dissolved the zinc from the baskets. Chemical examination showed the stewed fruit to contain 7 grains of zinc oxide per pound, and each person probably consumed an equivalent of 18 to 20 grains of zinc sulphate, whilst the emetic dose of this salt is from 10 to 30 grains. Recovery from the effects of the poison was complete by the following day, even in the case of those individuals most seriously affected.

T. J. W.

**Metallic Poisons in Foodstuffs. W. M. Willoughby.** (*Lancet*, 1923, **204,** 255.)—It is generally stated that the presence of tin in canned foods is due to the action of fruit or vegetable acids or meat extracts upon the tin coating of the containing vessel, but the author points out that de-tinning is most probable with those foods to which sodium chloride is added. Even "sanitary" containers, the interior of which is lacquered, are not free from electrolytic action, owing to cracking of the lacquer and sometimes of the tin coating in the process of crimping the lid. The electrolysis set up between the exposed metals and the salt solution not only contaminates the contained food, but also leads to the development of pinholes through which air gains access to the interior of the cans. The wood shavings or paper case surrounding canned lobster serves only to prevent discoloration of the fish and has no effect upon metallic contamination. T. J. W.

## Biochemical, Bacteriological, etc.

**Catalytic Destruction of Carnosine in Vitro. W. M. Clifford.** (*Biochem. J.*, 1922, **16,** 792–799.)—Minced lean beef muscle mixed with water and maintained at a temperature of 100° C. for several days showed a gradual diminution in carnosine content, equivalent in 3 weeks to the loss observed in 9 to 10 months at 0° C. (ANALYST, 1921, **46,** 507; 1922, **47,** 443). The catalyst to which this action is

attributed is not removed from beef by short extraction with hot or cold water, but by prolonging the extraction for 48 hours this substance is removed from the meat and passes into solution. Although carnosine is absent from the muscles of white fish, the catalyst was found to be present and produced degradation of the carnosine in beef extract. The catalyst occurs in various tissues of vertebrates, but is not found in the kidney, in tissues subjected to long-continued cold storage, nor in the tissues of the lobster or oyster. Curves showing the rate of action of the catalyst are unlike those of an enzymic reaction, and indicate little or no action during the first six days, but rapid destruction of carnosine during the succeeding two days, followed by a further period of two days in which little change is observed, after which the action recommences and proceeds slowly and steadily for 17 days. It is suggested that this behaviour may be due to the activity of two different agents, or of one catalyst acting upon carnosine in two stages. T. J. W.

**Vitamin *D*. T. B. Heaton.** (*Biochem. J.*, 1922, **16**, 800–808.)—The stimulating action of Wildier's "bios" upon yeast cells in small concentration is not yet understood, but recent researches have shown that the substance is organic, is soluble in water and in alcohol, dialyses readily and is thermo-stable. These characteristics indicate that the substance belongs to the vitamin group, although its stability to heat is greater than that of vitamin *B*. The estimation of "bios" has been carried out principally by determining its effect upon the rate of multiplication of yeast cells, or by measuring the amount of carbon dioxide evolved during fermentation. The author has adopted the latter method, using Nageli's solution from which the ammonium nitrate was omitted, and has tested the action of cow's milk, dried milk, dried yeast, calf spleen, various organs from normal and polyneuritic pigeons, and from normal rats and others fed upon a vitamin-free diet. The substance was found to be present to an equal extent in various organs of normal rats and of normal and polyneuritic pigeons, but progressively diminished in the rats fed upon a diet free from vitamins *B* and *C* to about half the normal amount at death. This distribution is indicative of the presence of a substance distinct from vitamin *B*, and this substance has been named "bios" by Wildier and vitamin *D* by Funk. (*Proc. Soc. Exp. Biol.*, 1921, **19**, 15.) T. J. W.

**Electrolytic Estimation of Lead in Biological Work. A. S. Minot.** (*J. Biol. Chem.*, 1923, 1923, **55**, 1–8.)—Owing to discrepancies between the results obtained by the Denis-Minot electrolytic method and the Fairhall chromate method (*J. Biol. Chem.*, 1919, **38**, 449, and *J. Ind. Hyg.*, 1922, **4**, 9) in the estimation of lead in biological material, the author has investigated the former process. It is shown that positive results are given in the absence of lead owing to the frequent presence of manganese which is deposited as the peroxide, this compound giving the same reactions as lead peroxide in the subsequent procedure. Losses of lead, when present, are attributed to partial solution of the sulphides during washing and incomplete electrolytic separation owing to the presence of phosphates in solution. The method is condemned as unreliable for either quantitative or qualitative

purposes, and the suggestion is made that improvement would result by precipitating the lead as sulphide in faintly acid solution. Several tables are given showing results obtained with pure solutions of lead and manganese salts and with ash obtained from fæces. Fairhall's method is shown to be reliable and to be simpler in manipulation than the electrolytic method. T. J. W.

# Water Analysis.

**New Method of Measuring Corrosion in Water Pipes. F. N. Speller and V. V. Kendall.** (*J. Ind. Eng. Chem.*, 1923, **15**, 134–138.)—The corrosive action of natural waters on iron pipes has been shown to be directly proportional to the amount of dissolved oxygen, and a method proposed for measuring the corrosion consists in estimating the dissolved oxygen before and after the water has been passed through an iron pipe under suitable conditions. The apparatus described is a coil about 18 inches in diameter of 0·25 inch iron pipe, placed in a drum; taps are inserted in the coil at various parts of its length, so that a constant time of contact can be maintained at different velocities. Means are provided for keeping the water at any desired temperature. During its passage through the coil the water is kept under pressure and is cooled, by passing through a brass coil, before the remaining dissolved oxygen is estimated. It is shown that corrosion increases with velocity of flow and with increase of temperature.

W. P. S.

# Agricultural Analysis.

**Microscopic Method for Estimation of Rice Hulls in Bran. B. H. Silberberg.** (*J. Assoc. Off. Agric. Chem.*, 1922, **6**, 71–72.)—The estimation of rice hulls in rice bran has been made on 5 samples by (*a*) deduction from the crude fibre value and (*b*) a microscopic method (*J.A.O.A.C.*, 1921, **5**, 77), with the following results:

| | Percentage of Hulls. | | | | |
|---|---|---|---|---|---|
| Sample | A | B | C | D | E |
| From crude fibre | 21 | 21 | 19 | 9 | 10 |
| By micro. method | about 20 | 20–25 | 15–20 | not over 10 | less than 10 |

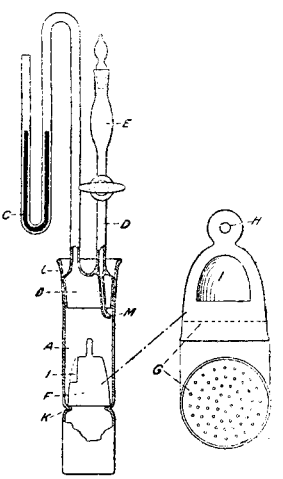
The recommendation is made that the microscopic method should be adopted as a tentative one by the Association. T. J. W.

**Respiration of Apple Seeds. G. T. Harrington.** (*J. Agric. Research*, 1923, **23**, 117–130.)—The respiration of apple seeds (Newtown Pippins) has been investigated by means of the respirometer described (see following abstract), and it is shown that the respiratory quotient $CO_2/O$ for dormant seeds at ordinary temperatures (19° C.) is 0·7, which corresponds to the complete oxidation of the fats or only a slight increase in sugars, but that the quotient rapidly increases with increase in temperature. The intensity of respiration of germinating seeds is greater than that of dormant seeds, but the quotient becomes lower, indicating rapid transformation of fats and accumulation of sugars, though there is sometimes

a brief initial rise, probably due to the oxidation of organic acids. The temperature coefficients are greater for dormant seeds which have been previously incubated at medium temperatures (20° C.) than for seeds which have been incubated at higher temperatures, and it is shown that the temperature coefficients are different in the different steps in the oxidation processes which constitute respiration. The investigation shows also that it is essential to study oxygen consumption in relation to carbon dioxide production for the elucidation of respiratory processes.

H. E. C.

**New Respirometer for Seeds and Other Small Objects. G. T. Harrington and W. Crocker.** (*J. Agric. Research*, 1923, **23**, 101–115.)—A review is given of previously described respirometers, and it is is shown that none is completely satisfactory. The defects are obviated in the apparatus (see figure), which has been designed to measure accurately both the carbon dioxide produced and the oxygen consumption on the whole volume of the air in a given period of time. The apparatus is made in three sizes, of 20, 40, and 80 c.c. capacity, and consists of a glass tube A fitted with stopper B carrying a manometer C of 2 mm. bore, and an inlet D with a bulb E to contain sodium hydroxide solution. Inside A is the seed container F, with its perforated bottom G and an opening I in the side and a ring on the top for raising or lowering it. A special carrier is also described for holding several of these respirometers. For use, the seeds are placed on G in the container F, and the apparatus is aerated by a rapid current of air, and, after insertion of the stoppers, is placed in a water bath at the desired temperature, and mercury is poured round the joint L. When the temperature is uniform the stop-cock is closed, care being taken that the height of the mercury in the manometer limbs is equal. The barometric pressure is now noted. After the desired time (usually 24 hours) from 1 to 3 c.c. of 20 per cent. sodium hydroxide solution is poured into E and the stopper left out; the manometer is now read by placing a scale behind it; and then the soda solution is run into the respirometer bulb but without wetting the seeds. After a short interval the manometer and barometer are again read. The volumes of oxygen absorbed, carbon dioxide given off, and the respiratory coefficient are readily derived from the changed position of the mercury after allowing for the volume of the seed and of the soda solution run in. Tables for abbreviating the calculations are given. H. E. C.




**Effects of the Method of Desiccation on the Carbohydrates of Plant Tissue. K. P. Link and W. E. Tottingham.** (*J. Amer. Chem. Soc.*, 1923, **45**, 439–447.)—Enzymic activity and respiration of plant tissues are liable to change the carbohydrates and proteins during drying. To find the most satisfactory

method of desiccation the following five types of plant tissue were dried by different methods and their carbohydrates estimated:—Beet leaves, maize leaves, maize stalks, maize seedlings, and potato tubers. The results indicate that a temperature of 98° C. should not be employed where there is much starch or sugar present; but if the tissue is readily reducible to thin sections they may be dried rapidly in an air current at 65° C., or preferably *in vacuo* at 80° C. Coarse tissues should be heated in an autoclave to inhibit enzymic action and then dried at 80° C. *in vacuo*. Similar results may be obtained by immersing the tissues in 95 per cent. alcohol in sufficient quantity to ensure that after allowing for the water in the tissues the alcoholic strength is not reduced below 75 per cent., and then heating for an hour at 78° C. Treatment with cold alcohol is unsatisfactory. None of the methods investigated is recommended when it is desired to estimate the soluble protein. H. E. C.

# Organic Analysis.

**Method for the Simultaneous Estimation of Sulphur and Halogen in Organic Compounds. C. S. Leonard.** (*J. Amer. Chem. Soc.*, 1923, **45**, 255–257.)—The following modification of Klason's method (*Ber.*, 1886, **19**, 1910) is described:—One end of a glass combustion tube is drawn out and bent to dip below the surface of 100 c.c. of water in a receiver flask. At the opposite end the tube is attached by means of a fume-treated cork to a bulb of sulphur-free, fuming nitric acid. A stream of dry air or oxygen may be passed through acid and tube. The tube contains, at the bent end, first a roll of freshly ignited, fine platinum gauze (A), and then a porcelain boat (B) filled with fuming nitric acid. At the midpoint a similar platinum gauze (C) is placed, a second boat (D) containing the substance to be analysed being pushed into the fore part of the tube after the gauzes have been heated to dull redness and air has been passed to fill the tube with brown fumes; a low flame is applied at or near the boat B in order to keep this portion of the tube filled at all times with the fumes. The tube is gradually heated near and ultimately under boat D, but some substances may oxidise without heating, as is shown by the appearance of white fumes. The heating and air current are so regulated that brown fumes are never absent beyond A, but white fumes may be permitted just beyond C if more nitric fumes are immediately supplied by application of a low flame to the nitric acid bulb and, if necessary, by increasing the heating below B. Finally, the material is driven along the tube by the application of heat, but red heat below the nitric acid boat must be avoided. Combustion must never be sufficiently rapid to produce flashes in the tube; there is most danger of this if, as is sometimes desirable with refractory substances, the combustion is carried out in oxygen instead of in air. After the combustion, which occupies 1½ to 2 hours, the boats and spirals are rinsed and finally boiled with water, and the walls of the tube rinsed with water into the receiver. The whole of the rinsings and the contents of the receiver are combined and, if sulphur alone is to be estimated, heated on a steam bath until all odour of nitric acid disappears, the sulphate being then precipitated as usual. For the estimation of halogen, a

blackened receiver is used containing a slight excess of silver nitrate solution. The silver halide is filtered on a Gooch crucible, washed with warm 2 per cent. nitric acid, and weighed; for halogen alone, Volhard's method may be used. The sulphur is estimated as usual in the filtrate from the Gooch crucible, the excess of silver being first removed by means of hydrochloric acid. About 0·1 to 0·2 grm. of substance is used for the combustion, 0·1 grm. sufficing when the percentage of sulphur present is 30 to 40. T. H. P.

**Separation and Identification of Alcohols by means of Bromomethyl-phthalimide. H. H. Hopkins.** (*J. Amer. Chem. Soc.*, 1923, **45**, 541–544.)—Bromomethyl-phthalimide forms with the aliphatic alcohols phthalimidomethyl esters, the melting points of which decrease with the increasing molecular weights of the alcohols, and so are suitable for the identification of alcohols. For the esterification, 5 grms. of the -imide are digested with about 10 grms. of the alcohol for 10 to 15 hours, the solution then concentrated and crystallised, and the melting point of the ester determined. Methyl alcohol can be separated from acetone in the form of its ester. The method has the disadvantage that the alcohols must be quite anhydrous, and moisture must be rigorously excluded during the esterification. The melting points of several phthalimido-methyl esters of the alcohols are given. H. E. C.

**New Hexose-phosphoric Ester. R. Robison.** (*Biochem. J.*, 1922, **16**, 809–823.)—Observations made by the author in 1913 while preparing hexose-diphosphoric acid from lævulose indicated the existence of a similar compound possessing a different composition (*Proc. Chem. Soc.*, 1914, **30**, 16). Recent research has resulted in the identification of this compound as hexosemono-phosphoric acid, and a detailed account of the preparation and properties of the acid, its salts and esters is given. The acid has an $(\alpha)_D^{20°}$ of +25·0°, and, on hydrolysis by acids or emulsin, yields phosphoric acid and a reducing substance which, with phenylhydrazine, produces dextrosazone, but has a rotatory power less than that of pure dextrose. The phenylhydrazine salt of the osazone of hexose-monophosphoric acid has a m.pt. of 139° C., and is therefore not identical with the compound having the same empirical formula prepared by Young and Lebedev (*Biochem. Zeitsch.*, 1909–1911). The normal salts of the heavy metals are amorphous, and readily soluble in water, the brucine salt being also soluble but crystalline, and the salts of the alkali metals are readily fermented by yeast juice and zymase. The acid is distinct from the hexosemonophosphoric acid of Neuberg (*Biochem. Zeitsch.*, 1918, **88**, 432) prepared by the partial hydrolysis of hexosediphosphoric acid. T. J. W.

**Use of Schiff's Reagent in the Quantitative Estimation of Acrolein. C. Moureu and E. Boismenu.** (*J. Pharm. Chim.*, 1923, **27**, 49–54, 89–97.)—Under certain conditions Schiff's reagent can be used for the quantitative colorimetric estimation of acrolein. The solution to be examined must be diluted to slightly below the strength of the type solution (1 in 2000), which is prepared by

distilling acrolein into a test tube containing a weighed bulb, fixed to the condenser by means of a cork in which is also a very small bored hole. When about 20 grms. of acrolein have been distilled the test tube is detached and rapidly exhausted, so that when air is subsequently admitted the acrolein enters the bulb, which is immediately sealed and weighed. The amount of water necessary to give a solution of 1 in 2000 is then calculated, and the bulb broken in the water so that the acrolein is dissolved. In order to have solutions of approximately the same strength, 5 c.c. of Schiff's reagent are added at the same time to 10 c.c. of the type solution and 10 c.c. of the unknown solution, and the colours compared. The Schiff's reagent is prepared by adding 50 c.c. of an aqueous solution of 1 grm. per litre of fuchsin and 250 c.c. of water to 20 c.c. of sodium bisulphite solution of 40° B., and, after mixing and partial decolorisation, adding 2 c.c. of concentrated sulphuric acid. The estimation is made by introducing 10 c.c. of each solution into one limb of two specially constructed Y-tubes, and 5 c.c. of Schiff's reagent into the other limb. After corking the tube the liquids are mixed at the same time, and, after 25 minutes, examined colorimetrically by finding the height of solution necessary to give the same depth of colour as 25 mm. of the type solution. A table is given showing concentration plotted against the height of the column, and when working with solutions of known strength the authors obtained accurate results, and found that a difference in concentration of 5 per cent. gave a difference of 3 m.m. in the height of the column. Since certain compounds (pyrogallol, pyrocatechol, hydroquinone, gallic acid, and tannin), in a proportion up to 1 per cent., have a stabilising effect on acrolein, and do not interfere with the colorimetric estimation, an addition of one of these renders it unnecessary to prepare a fresh type solution each time an estimation has to be made. D. G. H.

**Microchemical Detection of Fumaric Acid. L. van Itallie.** (*Pharm. Weekblad.*, 1922, **59**, 1312-1314.)—On treating a solution of ammonium fumarate with a little thallium nitrate the reagent dissolves, and colourless prismatic crystals are formed after some time. Better results are obtained by the use of lead acetate, which produces colourless prismatic plates or needles, not infrequently in the form of stars. Copper acetate gives light blue spheroidal masses or needles, grouped here and there into stars, and if the excess of ammonia is allowed to evaporate, blue prismatic crystals are produced. Particularly fine crystals are obtained by dissolving cupric acetate in ammonia solution, and mixing one drop of this reagent with one drop of the fumarate solution.

**Estimation of Water in Mineral Oils. L. Losana.** (*Giorn. Chim. Ind. Appl.*, 1922, **4**, 570-573.)—The estimation of water in a mineral oil by treating the oil with liquid sodium amalgam and measuring the volume of the hydrogen evolved gives good results for low, but is inconvenient for high, contents of water. The author finds that, no matter what the proportion of moisture in the oil, it may be estimated accurately by measuring, not the volume of the hydrogen, but the pressure required to keep the total gas-content of the apparatus constant.

Use is made of a pump-flask with a rubber stopper held down by a stirrup, the side-tube being connected by rubber tubing with the stop-cock of a manometer tube. The oil, mixed with a fixed quantity of dehydrated petroleum, is placed in the flask and the liquid sodium amalgam in a small test-tube supported in the flask by two wires passing through the stopper in such a way that, when the apparatus is connected up, the tube with the amalgam may be allowed to fall into the oil. The flask is then shaken and the pressure adjusted and read off on the manometer after it has remained constant for 10 minutes. If the volume of the flask and that of the oil and petroleum are known, the volume of hydrogen evolved may be calculated from the pressure generated, but a more convenient plan consists in determining the actual values of the pressure obtained with a number of prepared samples of oil containing known proportions of water. Calcium may be used in place of the sodium amalgam, but reacts more slowly. T. H. P.

**Analytical Characters of Commercial Linseed Oils. H. Wolff.** (*Chem. Zeit.*, 1923, **47**, 142–145.)—The following is a summary of the results obtained by the analysis of some three hundred samples of genuine linseed oil:—Sp. gr. at 20° C., 0·927 to 0·931; 88 per cent. of the results lay between the values 0·9280 and 0·9305. Refractive index at 20° C., 1·4785 to 1·4815, 65 per cent. of the values being between 1·4795 and 1·4810. Iodine value (Hübl–Waller), 170 to 192, 77 per cent. of the results being between 172 and 177 and 94 per cent. between 170 and 180. Saponification value, 187 to 196, with 86 per cent. of the results between 188 and 192. Yield of hexabromides, 50 to 59 per cent. W. P. S.

# Inorganic Analysis.

**Stability of Dilute Sodium Oxalate Solution. E. S. Hopkins.** (*J. Ind. Eng. Chem.*, 1923, **15**, 149.)—Dilute (0·01 *N*) sodium oxalate solution deteriorates when kept, losing about 25 per cent. of its reducing power (towards permanganate) in about four months. The addition of 100 c.c. of dilute sulphuric acid (1:4) per litre of the oxalate solution will prevent this loss of strength even when the solution is kept in clear glass bottles and exposed to daylight.

W. P. S.

**Electrolytic Estimation of Mercury. A. De Meeŭs.** (*Bull. Soc. Chim. Belg.*, 1922, **31**, 302–323.)—It is of distinct advantage when estimating mercury electrolytically to use a gold cathode instead of one of platinum, especially when the mercury is to be deposited rapidly. The electrode with the deposit may then be washed with alcohol and ether and dried rapidly, and weighed without the loss which always occurs if a platinum cathode is so treated. For the estimation of mercuric salts a quantity containing about 0·3 grm. of mercury is dissolved in dilute nitric or sulphuric acid and deposited on an electrode rotating at about 600 r.p.m. by a current of density 4 to 5 amps. at a temperature of 30° to 40° C., with this current the deposition is complete in about 20 minutes; or 1 to 2 amps. may be employed, in which case the deposition requires about 45 minutes.

Mercurous salts are brought into solution in dilute sulphuric acid by oxidation with a slight excess of potassium permanganate or dichromate solution. In the case of the iodide, however, this method cannot be used, but oxidation may be brought about in dilute nitric acid solution by means of bromine water. Mercurous sulphate or oxide are also preferably oxidised by the latter method, as the permanganate or dichromate oxidation is very slow. The use of sulphuric or nitric acid for the solution of the mercury compound is preferable to the employment of cyanides or iodides which form complex salts; their presence, however, does not invalidate the process. H. E. C.

**Volumetric Estimation of Mercury by means of Sodium Chloride. Analysis of Cinnabar and Organic Mercury Compounds. E. Votoček and L. Kašparek.** (*Bull. Soc. Chim.*, 1923, **33**, 110–122.)—When sodium nitroprusside is added to a solution of a mercuric salt in dilute nitric acid a turbidity due to mercuric nitroprusside is formed, and the mercury may be estimated by titrating the solution with a standard solution of sodium chloride until the turbidity disappears. The temperature should be about 15° C., and 0·06 grm. of nitroprusside should be employed for each 200 c.c. of liquid to be titrated; the (empirical) factor is then 0·010124 grm. Hg. for each c.c. of 0·1 *N* sodium chloride. For the estimation of mercury in cinnabar, 0·3 grm. of the finely ground mineral is digested in a Kjeldahl flask with 50 c.c. of a mixture of 2 volumes of sulphuric acid with 1 volume of nitric acid, until decolorised; the mixture is then diluted and any nitrous acid present is oxidised by the addition of potassium permanganate; excess of permanganate is removed by the addition of a few drops of a solution of oxalic acid, and an aliquot part is titrated as before. Aromatic organic compounds containing mercury are not readily decomposed by treatment with nitric and sulphuric acids; they are therefore brought into solution by digestion with 15 to 20 c.c. of nitric acid on the water bath, with the gradual addition of solid potassium permanganate until the colour of the permanganate is no longer destroyed. After cooling, the manganese is all dissolved by the addition of sodium nitrite solution, excess of nitrous acid removed by means of permanganate and excess of the latter by means of oxalic acid as before; the liquid is then diluted and an aliquot part titrated with 0·1 *N* sodium chloride. In the case of the non-ionising mercury halogen salts, the mercury is first precipitated with hydrogen sulphide, and the mercuric sulphide then digested with the mixture of nitric and sulphuric acids until dissolved; after which the solution is titrated. The presence of alkali or alkaline earth metals or of those metals, the nitroprussides of which are soluble in water (Pb, Zn, Al, Cr, Fe, Mn), does not interfere with the volumetric estimation by this method. H. E. C.

**New Method for the Electrometric Titration of Vanadium in the Presence of Iron and Chromium. H. H. Willard and F. Fenwick.** (*J. Amer. Chem. Soc.*, 1923, **45**, 84–92.)—The electrode system previously described (*ibid.*, 1922, **44**, 2516), consisting of two identical platinum wires polarised with a current of $0{\cdot}5\times10^{-5}$ ampère, may be applied to the electrometric titration of

vanadic acid with ferrous sulphate. The voltage rises slowly from the first addition of reducing agent, and more slowly as the concentration of vanadyl salt increases. Just prior to the end-point, this velocity increases very slightly and then decreases again, and at the end-point the galvanometer appears momentarily to have lost its sensitiveness, and then reverses the direction of its swing as more ferrous sulphate is added. In carrying out a titration it is best to follow the rising voltage with the potentiometer, keeping about 0·5 millivolt behind it. The reversal is very sharp, easy to detect and normally sensitive to 0·03 c.c. of 0·02 *N* solution.

To estimate the vanadium in vanadium, chromium-vanadium, and molybdenum-chromium-vanadium steels, a weighed quantity requiring about 10 c.c. of 0·02 *N* ferrous sulphate solution is placed in a 600 c.c. beaker with 20 to 30 c.c. of water, and a measured volume of concentrated sulphuric acid run in from a burette; each gram of steel requires 1 c.c. of the acid to form ferrous sulphate, and an excess of 4 c.c. suffices to effect rapid dissolution. The liquid is heated gently until the metal is completely dissolved, and the salts begin to separate, 20 c.c. of hot water being then added and the heating continued until solution is complete. The solution is then treated cautiously with 4 or 5 c.c. of concentrated nitric acid and boiled, oxidation of the iron and vanadium being completed by means of slight excess of potassium permanganate solution. Sufficient sodium acetate to combine with the acid used in excess of that required for solution (1 c.c. of concentrated sulphuric acid corresponds with 4·8 grms. of trihydrated sodium acetate), 40 to 50 c.c. of glacial acetic acid, and 0·5 grm. of sodium perborate, previously neutralised, are added, the liquid being diluted, if necessary, to 200 c.c. and boiled for 20 minutes. The solution is cooled to room temperature, treated with 25 to 30 c.c. of concentrated hydrochloric acid, and titrated with 0·02 *N* ferrous sulphate solution standardised upon potassium dichromate.

In the case of chromium-vanadium-tungsten steel, the sample is heated with 40 c.c. of hydrochloric acid (3 parts of the concentrated acid to 1 part of water) until action ceases, 8 to 10 c.c. of concentrated nitric acid being then added, dropwise, until the first violent action has ceased. The liquid is boiled, evaporated to about 20 c.c., diluted with hot water, heated to ensure complete solution of soluble salts, and filtered, and the filter washed with hot 2 per cent. hydrochloric acid solution. The filtrate is oxidised with permanganate solution and treated with sufficient sodium acetate to combine with the free acid; this may be estimated accurately enough by taking the weight of hydrochloric acid as equal to 20 per cent. of the volume after the solution is concentrated to about 20 c.c. and adding 5 to 6 grms. more as a safeguard (1 grm. of hydrochloric acid corresponds with 3·73 grms. of trihydrated sodium acetate). The subsequent procedure is as described above. To determine the vanadium occluded by the precipitated tungstic acid, this is dissolved in sodium carbonate solution, a few decigrams of perborate being added and the liquid boiled vigorously for 10 minutes, acidified with 3 to 5 grms. of phosphoric acid, treated with 25 to 40 c.c. of sulphuric acid (1 part of acid to 3 parts of water), and titrated with ferrous sulphate solution electrometrically to the permanent drop in potential.

If tungstic acid is dissolved in sodium carbonate solution and the solution acidified with phosphoric acid, treated with dilute sulphuric acid and a known amount of ammonium vanadate, and the mixture titrated with ferrous sulphate as above, the character of the end-point is found to be altered materially by the presence of the phosphotungstic acid. As titration progresses, the solution assumes a deep amethyst colour, probably owing to the formation of a vanadyl phosphotungstate, and at some distance from the end-point each drop of ferrous sulphate solution causes a fall in the potential, followed by a slow return to the former maximum. The first decrease in voltage lasting for two minutes or longer may be assumed to be permanent and accepted as the true end-point. In presence of phosphomolybdic acid, the method gives rapid and accurate results, provided that hydrochloric, and not sulphuric acid, is added. This method may be applied to the direct titration of the vanadium occluded by the phosphomolybdate precipitate (*cf.* Cain and Hostetter, ANALYST, 1922, **47**, 184). T. H. P.

**Quantitative Separation of Beryllium and Uranium. P. H. M. Brinton and R. B. Ellestad.** (*J. Amer. Chem. Soc.*, 1923, **45**, 395–398.)—A review of published processes has shown that no satisfactory method has yet appeared for the separation of beryllium and uranium. The separation can, however, be accurately effected by precipitating the beryllium by ammonium carbonate in the presence of hydroxylamine hydrochloride, and removing the last trace by means of ammonium hydroxide. The precipitate obtained in this way is easily filtered and washed free from uranium. To the hydrochloric acid solution of the metals is added about 5 grms. each of ammonium chloride and of hydroxylamine hydrochloride, followed by a concentrated solution of ammonium carbonate until the precipitate redissolves; the liquid is boiled and the precipitate formed is filtered off and washed with cold water. The filtrate is acidified, boiled, cooled, and a further 1 grm. of hydroxylamine hydrochloride is added and then excess of ammonia. The small precipitate which forms is collected and washed with a 2 per cent. solution of ammonium nitrate containing a few crystals of hydroxylamine hydrochloride and enough ammonia to render it just alkaline. The combined precipitates are then ignited with the papers and weighed as BeO. For the estimation of the uranium in the filtrate the hydroxylamine is oxidised by boiling with hydrogen peroxide or sodium bromate, and the uranium precipitated by adding excess of ammonia; the precipitate is boiled, filtered, washed with 2 per cent. ammonium nitrate solution (not containing any hydroxylamine) and weighed as $U_3O_8$. H. E. C.

**Analysis of Sodium Perborate. H. Burkardt.** (*Chem. Zeit.*, 1923, **47**, 6.)—The oxygen-content of this salt is estimated by titration with 0·25 $N$ permanganate solution in presence of sulphuric acid; if 0·975 grm. of the salt is taken, the number of c.c., say $b$, of permanganate used represents the percentage of $Na_2O_2$. The borate-content is ascertained by using $N/2$ sodium hydroxide standardised with methyl orange as indicator. The carbonate content of this solution is indicated by a factor $k$; thus, if 10 c.c. of the 0·5 $N$ sodium hydroxide neutralise

9·5 c.c. of 0·5 *N* hydrochloric acid, with phenolphthalein as indicator, $k=0{\cdot}95$. For the estimation 2·5 grms. of the salt are dissolved in water (which need not have been rendered oxygen-free by boiling), and the solution neutralised towards methyl orange by means of *z* c.c. of 0·5 *N* hydrochloric acid, any carbon dioxide derived from carbonate or percarbonate being eliminated by boiling the liquid for 5 minutes under a reflux condenser; after cooling, the condenser is washed down and removed. The liquid is then mixed with 10 c.c. of neutral glycerol and titrated to a red colour with the 0·5 *N* sodium hydroxide solution in presence of phenolphthalein, a further quantity of 10 c.c. of glycerol being added and the titration continued; this procedure is repeated until addition of glycerol fails to decolorise the solution. If the total volume of 0·5 *N* sodium hydroxide required is *m* c.c. and *km* is represented by *y*, equality between *z* and *y* indicates that all the alkali in the sample is combined with boric acid, so that, besides perborate (and possibly borate), no salt having an alkaline reaction, such as percarbonate, can be present. The following calculations are then made:

| | |
|---|---|
| $z\times0{\cdot}62=t$ per cent. of $Na_2O$. | $b\times0{\cdot}397=d$ per cent. of $Na_2O$. |
| $y\times0{\cdot}7=u$ ,, ,, ,, $B_2O_3$. | $b\times0{\cdot}45=a$ ,, ,, ,, $B_2O_3$. |
| $y\times3{\cdot}08=v$ ,, ,, ,, $NaBO_3,4H_2O$. | $b\times1{\cdot}98=e$ ,, ,, ,, $NaBO_3,4H_2O$. |

From these it follows that:

$u-a=w$ per cent. of $B_2O_3$ present, not as perborate, but as borate.

$w\times2{\cdot}73=r$ ,, ,, ,, $Na_2B_4O_7,10H_2O$.

$w\times0{\cdot}442=s$ ,, ,, ,, $Na_2O$.

$t-d=s$, if all the oxygen exists as perborate.

$t-d$ is greater than *s*, if per-salts other than perborate are present.

T. H. P.

# Physical Methods, Apparatus, etc.

**Apparatus for Drying Organic Substances. J. Bouillot.** (*J. Pharm. Chim.*, 1923, **27**, 23–28.)—The apparatus consists of a horizontal glass tube, about 15 cm. in length and 3 cm. in diameter, closed at one end, and provided with a solid indiarubber stopper at the other. Two short narrow tubes are fused into the upper side about 5 cm. from either end of the horizontal tube, each of which is connected with the lower end of a vertical tube slightly smaller than the horizontal tube. The open ends of these tubes are fitted with indiarubber stoppers, through which pass narrow glass tubes drawn out to capillary bore at the lower ends, these ends being bent upwards. The lower ends of the vertical wide tubes are lightly plugged with cotton-wool. The substance to be dried is placed in a combustion boat and this inserted in the lower horizontal tube, which is then closed. The apparatus is suspended in an air-bath heated to the necessary temperature, the exit tube being connected with a filter pump, whilst the inlet tube is attached to one or more wash bottles containing sulphuric acid. By regulation of the air inlet desiccation may proceed under reduced pressure, or a current of inert gas may be used for readily oxidisable substances. The apparatus is easily manipulated and has been successfully employed for the dehydration of alkaloidal salts.

T. J. W.

# Reviews.

OPTICAL METHODS IN CONTROL AND RESEARCH LABORATORIES. By J. N. GOLDSMITH, S. JUDD LEWIS, and F. TWYMAN. Vol. I. (Spectrum Analysis, Absorption Spectra, Refractometry, Polarimetry.) Second Edition. Pp. 56. London: Adam Hilger Ltd. 1923. Price 1s. 6d.

This little book, although produced for trade purposes, is a valuable brochure on optical methods as applied in a technical laboratory; it is free from "puffing" or laudatory expressions, and, on reading it, one is impressed with the increasing usefulness of optical methods for industrial purposes. The refractometer and the polarimeter in some form or other have been found in every laboratory for many years past, but spectrometers or spectrophotometers have been generally considered as equipment only required in research or academic laboratories; the authors have drawn attention to the great utility of such apparatus, which affords a simple and rapid method for the quantitative as well as qualitative examination of very diverse materials. It is evident that a spectrograph is now a necessary instrument in the equipment of every up-to-date laboratory.

Different sections of the book deal with the metallurgical, analytical, and biochemical applications of the spectrometer, and the chemical and biochemical applications of the refractometer and polarimeter. As a means for the detection of minute quantities of elements present as impurity or otherwise the spectrometer is unrivalled, whether arc spectra or absorption spectra are employed, and the photographic method enables permanent records to be kept and the observations to be made by a laboratory attendant, so that the plates can be examined by the chemist at his convenience. Among the uses of absorption spectra may be mentioned the estimation of carbon monoxide in blood, the identification of alkaloids, the examination of glasses and the analysis of dyestuffs. The uses of the refractometer and polarimeter are well known, but by collecting the work of various investigators the authors indicate many fresh directions in which it may profitably be employed. New spheres of usefulness are also pointed out for the spectrometer, which has already proved such a powerful weapon of research in pure chemistry, as is testified by the discovery of the new element recently announced.

There are also useful data in the appendices to the volume, and the only thing lacking is an index. In short, the book is useful and valuable, and is commended to the notice of chemists generally. H. E. COX.

RESEARCHES ON CELLULOSE. Vol. IV. (1910–1921). By CHARLES F. CROSS and CHARLES DORÉE. Pp. 248. London: Longmans, Green & Co. 1922. Price 15s net.

The appearance of Vol. IV. of Cross and Bevan's *Researches on Cellulose* is marked by a change in collaborative authorship, owing to the death of Mr. E. J. Bevan, and by the fact that this volume has to cover a period of eleven years instead of the usual five years of the previous volume. Nevertheless, seeing that

the hand and brain of C. F. Cross remain at the helm, students and research workers in the field of "cellulose" need fear no lack of stimulating and imaginative food for thought in the pages of the present volume. This appreciation is justified in the opening chapter in which the construction and outlook of the survey are defined. The accumulation of research matter published in the past decade is discussed under four sections, each with its own peculiar perspective, namely: Cellulose as an organised colloid structure; Cellulose as a chemical, molecular individual; Cellulose under transformation, such as destructive distillation or fermentation; Cellulose in the service of industry. Of these four aspects it may be said, after perusal of this volume, that the first is the most interesting and enticing; the second has attracted most active attention and yielded the most positive results; the third is only derivative, coming into operation when the cellulose is no longer cellulose, and the fourth is too vast to be treated adequately in the space available; lastly, that all four are closely interdependent.

The skeleton of the book is constructed of abstracts of the more notable published researches classified according to the scheme just mentioned. These concise abstracts in themselves constitute an indispensable aid to the research worker, particularly since some of them are culled from literature relatively inaccessible to workers non-resident in the centres of learning. The special character of the book, however, is conveyed more definitely in the intermediate comments and the unpublished research matter which connect the various abstracts, placing them in their correct perspective and indicating whence they derive and whither they tend. In dealing with cellulose as an organised colloid the authors rightly dwell on the importance of its physical properties and relationships. A large accumulation of data on volume changes under different conditions reveals the fact that there is some underlying clue, but the time is not yet ripe for drawing the major theoretical conclusions, nor for appreciating at their true significance the various abnormalities recorded. A vast amount of work still remains to be done in studying the phenomena of hydration and co-ordinating their various manifestations. Hydration is one of the outward and visible properties of the colloid state, but up to now its manifestations along different paths remain on the empirical plane. It is good to see that the authors have not hesitated to reproduce in full the extremely significant research of H. E. Williams on the action of thiocyanates and other deliquescent salts from the *Manchester Memoirs*.

The period covered by this volume unfortunately just precludes a discussion of the most modern conception of the cellulose structure which is being developed by Continental workers, based on the crystal symmetry of the colloid so long as it remains "organised." The initial steps towards this vital hypothesis are noted in the form of a short "stop-press" abstract of Herzog's paper on the Röntgen Spectrographic investigation of Cellulose, but it appears without comment by the authors.

The chapter on the systematic constitution of cellulose as a chemical molecule is very ably compiled, and the story is well set out, even though the inevitable conclusions are not in harmony with the authors' well-known views. Chapter IV.,

Cellulose as an Organic Complex, should have come within the first section delimitated in the introductory classification, since it deals mainly with physical hydration phenomena induced by chemical means. In the section "Oxycellulose and Hydrocellulose" a number of investigations have been noted covering the action on cellulose of ozone, ultra-violet rays and hydrolysing acids. The theoretical discussion bears mainly on the nature of the association between the cupric reducing portion of the modified product and the main residue, which some observers rank as unaltered cellulose. This subject is still in the argumentative stage, with the balance of opinion tending towards the view that oxycellulose and hydrocellulose are not of homogeneous composition, but are adsorption compounds of unmodified cellulose, with variable proportions of its modified products. That the cellulose residue, although chemically indistinguishable, is really unchanged, appears open to doubt, and it is here that the distinction between organised cellulose and disorganised cellulose may find one of its first applications.

The section on lignocelluloses and lignone collects material from a widely dispersed field. The subjects under this heading comprise the following: Chemical and analytical constants of raw lignified materials; systematic chemistry of lignin (or lignone); nature of the celluloses isolated from lignocelluloses. The view that the cellulose nucleus of celluloses prepared from lignocelluloses is essentially identical with the typical cotton cellulose, due allowance being made for modification induced by the processes of treatment, has gained much ground in recent times. The acceptance of the essential original identity of celluloses, of all types, involves the recognition of the existence of a range of polymerised pentosans comprising members having a degree of resistance but little inferior to that of cellulose itself. Concerning cutocelluloses, a difficult subject only imperfectly explored, the authors have reprinted *in extenso* the very important research on raffia published by Cross and Bevan in 1919. The synthesis of cellulose esters of the higher fatty acids falls outside the period covered by this book.

The final chapter on "Cellulose Industries and Technology" is valuable from the fact that it contains accounts of certain researches of potential industrial interest which otherwise are not readily accessible; this remark applies especially to the action of micro-organisms on cellulose in various directions. The chapter, however, is not, as it might have been, a co-ordinated survey of the trend of research in the principal cellulose industries, and it is difficult to see how the perspective of the subject justifies the extensive reprint of the article by Cross and others on the use of gum tragasol in tanning.

Of the book as a whole, we again repeat that the most stimulating portions are the interspersed comments representing the personal view-points of the authors. In the conduct of research it does not matter whether the view-points are right or wrong; provided they exist at all, it is only continuous systematic work which counts in the end. To those who agree, and to those who do not agree, this volume is equally valuable if it leads to work in confirmation or refutation of the hypotheses formulated. The axiom still holds for the research worker, as for the logician, that a relationship must be assumed to be simple until it has been proved to be complex.

The subject is still full of gaps; and although this survey is only recently published, a lot of valuable material has since been built into those gaps. This fourth volume of "Researches" is a milestone and a finger-post erected by a master of the craft for those who are following the same road. We look forward to 1926!

J. F. BRIGGS.

BOILER FEED WATER. By PERCY G. JACKSON, F.I.C. Second Edition. Pp. xi.+143. London: Chas. Griffin & Co., Ltd. 1922. Price 5s. net.

The author's experience, as chemist to the National Boiler and General Insurance Co., has given him unique opportunities for acquiring an intimate practical knowledge of his subject, and the demand for a second edition of his book within two years is good testimony to its utility.

The aim of the work is strictly practical, and it appeals alike to the chemist and the engineer. It sets forth in the clearest possible manner the different ways in which feed waters may give trouble, and deals seriatim with the mineral constituents usually found in such waters, and the part, if any, which they may play in causing corrosion, priming or scale formation. The various methods of softening are described, typical forms of plant for this purpose are excellently illustrated, with useful hints on the selection of suitable apparatus for special purposes; and there are chapters on boiler control, priming, and on scale and its effects. Finally, some 30 pages are devoted to the analysis of waters and scales, the calculation of the quantities of softening materials required, and simple tests for ascertaining the efficiency of softening processes.

The chapters on the mineral constituents of waters, and the part they play in corrosion or scale formation, have been very carefully written, and the author's views, while perhaps conventional, are expressed with caution, and are such as few would cavil at. Controversial and speculative matters are avoided; and while the chemist may regret this, the engineer will not. A fuller treatment of the causes of corrosion and of the mechanism of scale formation, as well as some references to recent literature, however desirable from the purely scientific point of view, would have been impossible without detracting from the practical character of the book, and would have greatly increased its size. All the author's remarks are strictly to the point, and the only criticism we are inclined to make is that more stress might have been laid on the effects of pressure (*i.e.* of temperature) on the chemical reactions which are the prelude both to corrosion and the formation of scale.

In the sections on analysis the methods adopted, which make no claim to novelty, are described in detail, the composition of the reagents is given, and the methods of calculating the results and of allocating the various acids and bases to one another are fully explained. We cannot help feeling that the author is somewhat optimistic when he writes that a complete analysis of water, apparently including all the ordinary estimations except that of the alkalis, "can be comfortably carried through in an eight-hour day, with some spare time between operations," and that a batch of four can be completed in two days.

In view of the speed contemplated it is surprising that volumetric methods for the estimation of lime and magnesia have not been recommended, as the accuracy of those described by Burgess (ANALYST, 1907, **32**, 208) and Anderson (*J. Soc. Chem. Ind.*, 1915, **34**, 1181) is ample for this sort of work.

For the benefit of the chemist the brief references to the different types of oil-eliminating plants might with advantage have been somewhat more detailed, but on the whole the balance between the different topics has been very well maintained.

The Appendix contains a table of factors for the analytical calculations, useful formulæ for ascertaining the saline concentration likely to occur in boilers in a given time, and a table giving the capacity of cylinders of known diameter. Taken as a whole, the book is in every way excellent. In spite of its small size, every important aspect of the subject is dealt with; and while it is essentially practical, the theoretical side of the matter has received very clear and adequate treatment.

C. H. CRIBB.

## Journal of Scientific Instruments.

IT will be remembered that some time ago the Institute of Physics took steps to establish a Journal devoted to the description of methods of measurement and the construction and use of instruments in all branches of scientific and technical work.

As a first step the Institute published a preliminary number in order to ascertain how much support could be expected for such a Journal. The result was most encouraging, and the need for a Journal of the proposed nature was clearly manifest. This need has long been recognised in Germany, and, more recently, France and America have produced Journals similar to the one contemplated.

Arrangements have now been made with the Department of Scientific and Industrial Research for the Journal to be edited by the National Physical Laboratory, and the experience gained by the issue of the preliminary number has shown that the cost of production will be considerably less than was originally anticipated. There will, however, probably be a deficit during the first few years of the Journal's existence, and in these circumstances the Institute has decided to proceed with the project if it can be assured of a total guarantee fund of £1000, to be called up in instalments as required.

The Society of Public Analysts has been invited to contribute to the guarantee fund, but the Council feel that it would be outside the scope of the Society to make any monetary grant towards this end. The Council desire, however, to help forward the project in all ways possible, and it has therefore offered the Institute of Physics the publicity of its Journal in order to bring the matter before our members.

Some £650 has already been subscribed, and it is expected that the remainder of the amount required will be made up of comparatively small sums subscribed by scientific workers who realise the urgent need for such a Journal, if our scientific work and our manufacturing enterprises are to be developed with the highest possible efficiency.

Promises to subscribe to the guarantee fund will be gratefully received by the Secretary of the Institute of Physics, 10, Essex Street, Strand, W.C.2., and he will be glad to send a specimen copy of the Journal to any member who has not yet seen it.